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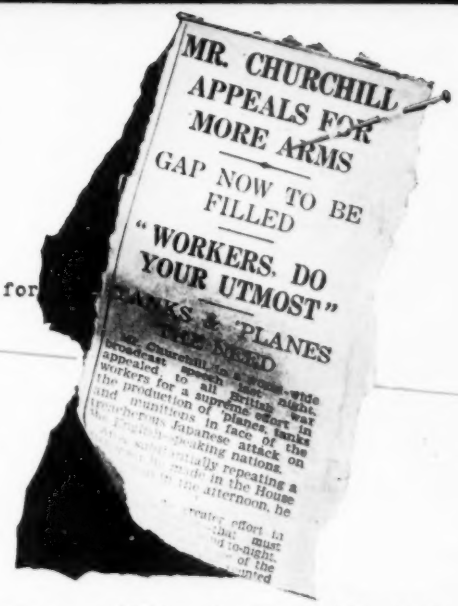

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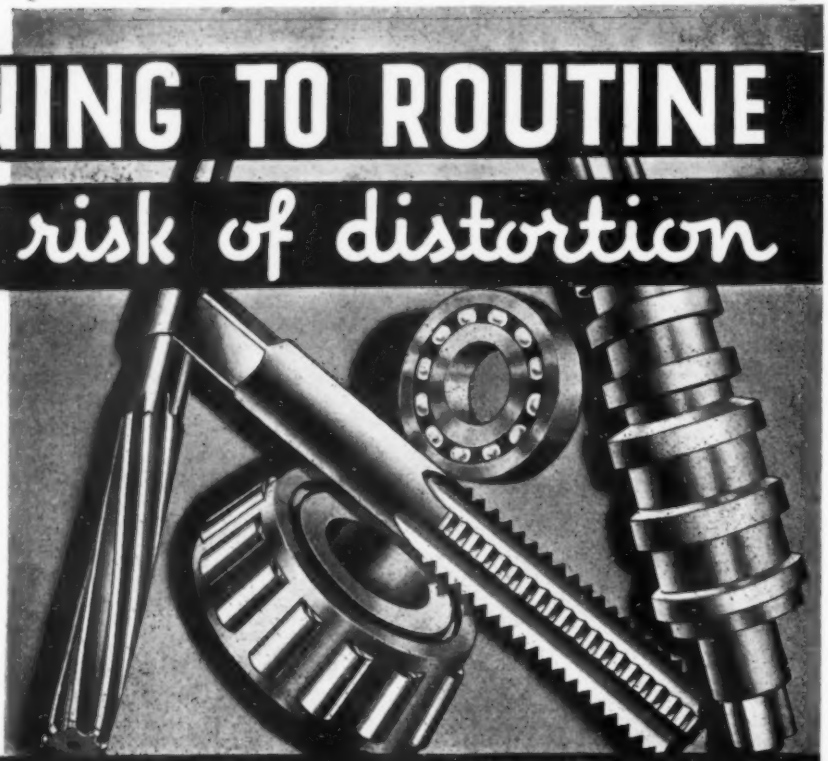

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JUNE, 1942. VOL. XXVI, No. 152.

Review of Recent Research into the Influence of Vanadium and Molybdenum upon the Creep Strength

By D. W. Rudorff, A.Am.I.E.E., M.Inst.F.

An investigation on the influence of the carbon content upon the properties of steel containing varying percentages of vanadium and/or molybdenum, and also silicon, is discussed. In addition to the examination of creep strength, tests were conducted to determine tensile strength, yield point, elastic limit, elongation and reduction in area, the results of which are given.

HEAT-RESISTANT steels alloyed with vanadium or molybdenum have found wide-spread application in many fields. Although as far back as in 1927 C. Wallman and F. Nehl suggested the employment of a low-alloyed steel with up to 0·3% Mo for parts subjected to high temperature,[1] present-day knowledge regarding the creep strength of Fe-C-V and Fe-C-Mo alloys still suffers from lack of systematisation, and empirical data are still quite incomplete, particularly as most investigations to date were concerned with the exploration of the high-temperature creep strength of steels with low carbon content.

In order to fill this gap a thorough investigation was recently undertaken by W. Holtmann[2] into the influence of the carbon content upon the properties of steel containing varying percentages of vanadium and/or molybdenum, and also of silicon. These tests were conducted at 20°, 500° and 550° C. In addition to the examination of creep strength investigations were also made of tensile strength, yield point, elastic limit, elongation, and reduction in area. These tests covered the following materials :—

(1) Steels with carbon contents ranging from 0·02 to 0·35%, and vanadium contents of 0·3 to 4·5%.
(2) Steels with carbon contents ranging from 0·02 to 0·5%, and molybdenum contents of 0·3 to 5·25%.
(3) Steels containing 0·02 to 0·4% C, 1·6% Mo, and 0·6% V.
(4) Steels with 0·02 to 0·42% C, 1·25% Mo, and 0·6% V.

The test-pieces, which were 18 mm. square, were pre-heated for 2 hours at 700° C. and then heated to hardening temperature. They were held at the latter temperature (not exceeding 1,100° C.) for ½ hour and then quenched in water, and finally tempered at 650° C. for 2 hours. The results of the various tests were charted in most instructive manner by means of the "topographical" method. In these charts (reproduced in Figs. 1—4) the tensile or creep strength values are given in the form of contour lines.

Referring to Fig. 1, it is seen that a maximum tensile strength of 113 kg./mm.² occurs with an alloy with a carbon content of slightly more than 0·3%, and a vanadium content of 1·3 to 1·5%. As the contour lines indicate, the decline in tensile strength from its maximum value takes place in a fairly gradual manner until a value of 85 kg./mm.² is reached. From this contour line onwards the tensile strength decreases rapidly to 40 kg./mm.², the influence of the decreased carbon content being particularly pronounced. In the region of high-vanadium and low-carbon percentage, variations in the percentage of carbon and vanadium are

[1] German Patent 552128 of February 18, 1927.
[2] *Stahl und Eisen*, vol. 62, 1942, p. 32.

Fig. 1.—Tensile strength of heat-treated vanadium steels at room temperature (20° C.).

[The numerals at the points through which the contour lines are drawn denote the tensile strength in Kg./mm.²; thus: "30" indicates a tensile strength of 30 kg./mm.² at the point concerned.]

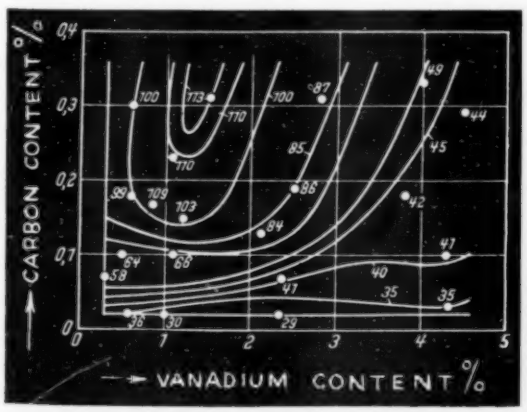
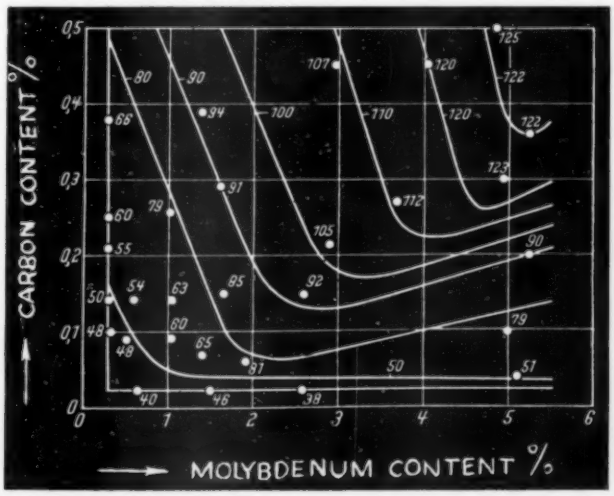


Fig. 2.—Tensile strength of heat-treated molybdenum steels at room temperature (20° C.).

[The numerals in the diagram denote values of tensile strength in kg./mm.²]



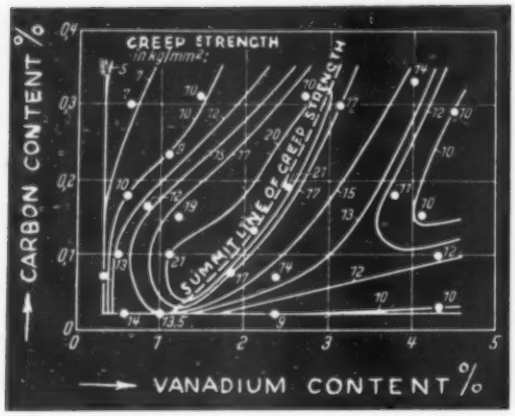


Fig. 3.—Creep strength of heat-treated vanadium steels at 500° C.

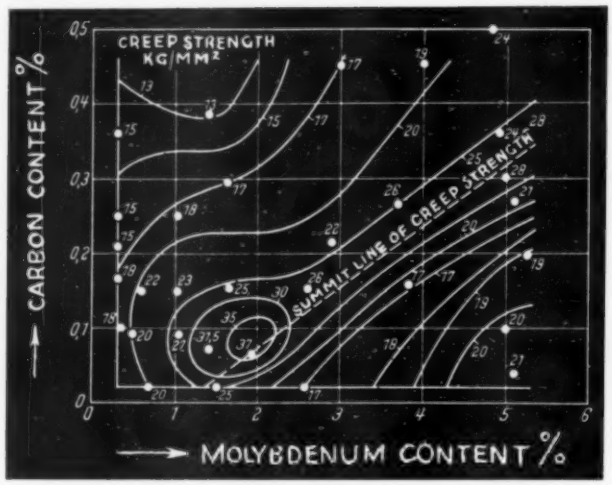


Fig. 4.—Creep strength of heat-treated molybdenum steels at 500° C.

seen to have but little influence upon the tensile strength. With regard to the influence of molybdenum content upon the tensile strength, Fig. 2 indicates that at room temperature the variation in tensile strength with varying molybdenum content is comparatively larger than that produced by a corresponding variation in vanadium content. Maximum tensile strength is seen to prevail in a region characterised by a carbon content in excess of 0·3%, and a molybdenum content exceeding 5%. As the contour lines indicate, the decline in tensile strength is more pronounced with diminished carbon content than with decreased molybdenum content. Both shape and relative disposition of the contour lines indicate that a certain relationship exists between the two alloying elements with regard to their combined effect upon tensile strength.

The contour diagram of the creep strength of the vanadium steels reproduced in Fig. 3 shows an extended region in which a creep strength in excess of 20 kg./mm.² at 500° C. is encountered. This region extends from about 0·05% C. and 1·2% V to 0·3% C. and 3·0% V. Near the borders of this region a more or less rapid decline in creep is registered, while a line of maximum creep strength traverses the length of the region. This "summit line" is seen to extend without change in direction beyond the field of high creep strength into the regions of lower creep strength. A similar line of maximum creep strength is seen to be drawn in the Mo-C creep strength contour diagram reproduced in Fig. 4. In this case the line of highest creep strength is seen to begin at 0·02% C and 1·5% Mo, and extends diagonally through the system towards the region signified by 0·35% C and 5·3% Mo. This line has a peak indicating particularly high creep strength of 37 kg./mm.² at a point represented by 0·06% C and 1·9% Mo content.

From this point a steep decline in creep strength is seen to take place in all directions. Also the disposition and configuration of the contour lines make it quite evident that maximum creep strength is associated with a definite molybdenum and/or vanadium content. Vice versa, definite vanadium or molybdenum contents must be co-ordinated with given carbon contents in order to obtain maximum values of creep strength.

A most interesting interpretation of the contour diagram is based on the fact that in iron-vanadium and iron-molybdenum systems the γ face centred cubic solution is only stable within the so-called γ-loop, and that the limit content of the alloying element increases with increased carbon content. This variation in limit values is charted in Fig. 5 and Fig. 6, respectively, where these limit values are seen to be represented by the limit curves shown.

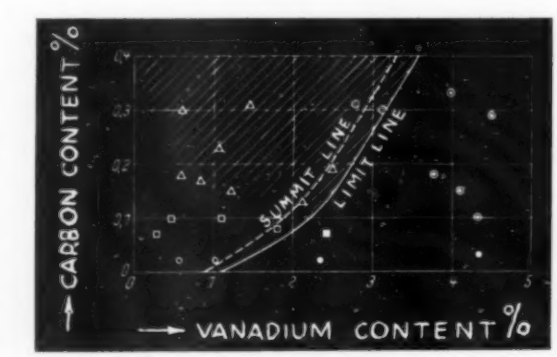
Obviously the steels ranged to the left of this curve according to their composition have undergone a complete $\gamma \rightarrow \alpha$ transformation; and in the range of low carbon and molybdenum or vanadium percentage their structure is composed of a mixture of martensite and ferrite. An increase in carbon and molybdenum or vanadium percentage results in a rapid decrease of ferrite content in favour of martensite content until a region characterised by martensitic structure is reached, these regions being shaded in Figs. 5 and 6. To the right of the limit curve untransformed ferrite is encountered owing to the occurrence of the γ-loop. Within the region of highest vanadium content a ferritic structure is encountered containing coarse carbides in the range of high carbon percentage.

With regard to the molybdenum steels with which the purely ferritic range is not reached, it should be noted that slow tempering at 650° C. of the steels containing 0·02% C and 2·6% Mo, and 0·1% C and 5% Mo, results in the precipitation of a finely grained component. In accordance with Houdremont's and Schrader's findings[3] this component was interpreted as Fe_3Mo_2. At and above 0·2% C the steels of classes (3) and (4) consist of pure martensite. Owing to the fact that their critical cooling speed is too high, steels with lower carbon percentage consist of a mixture of martensite and ferrite. With 0·02% C untransformed ferrite is encountered.

By incorporating in Figs. 5 and 6 the "summit lines" of maximum creep strength shown in Figs. 3 and 4, it is seen that these lines are approximately equidistant and equiform to the limit lines separating the region of complete transformation from that of incomplete or altogether

[3] *Techn. Mitt. Krupp, A: Forsch. Ber.*, 2 (1939), p. 23.

Fig. 5.—Structure, summit line of creep strength, and limit line of the ternary system Fe-V-C.



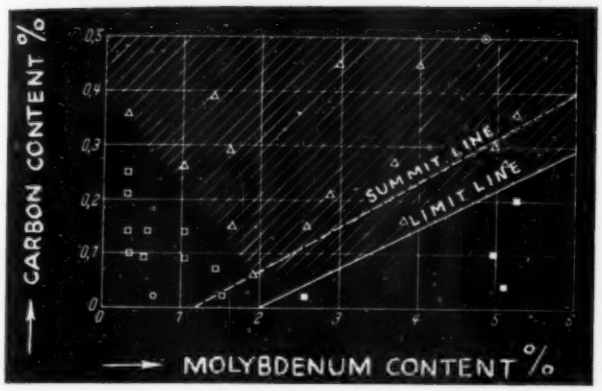


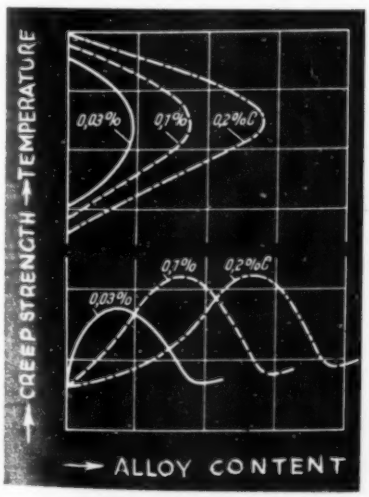
Fig. 6.—Structure, summit line of creep strength, and limit line of the ternary system Fe-Mo-C.

KEY
- ○ Ferrite
- △ Martensite
- □ Martensite + ferrite
- ⊙ Martensite + carbide
- } Quenched from γ-phase
- ■ Ferrite + martensite: Quenched from α or (α + γ)-phase respectively

absent α/γ transformation. If, for the sake of simplification, it were permissible to assume these two lines to coincide, the following inter-relationship between creep strength, tensile strength and structure could be established: In the steels ranged to the left of the limit line, consisting of a mixture of martensite and ferrite, the diminution in critical cooling speed caused by an increase in carbon and molybdenum or vanadium content results in a disappearance of the ferrite, and this leads to a considerable increase in creep strength and tensile strength.

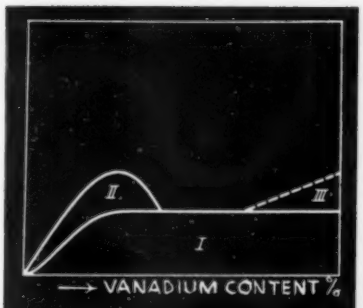
But when the region of purely martensitic steels is reached only a slow further increase in tensile strength with increased carbon and molybdenum or vanadium content takes place. In the case of the vanadium steels, the approach to the summit line with mounting vanadium percentage is found to lead even to a diminution in tensile strength; this diminution was found to be due to the presence of undissolved vanadium carbides. In the martensitic region the creep strength showed an increase with augmented vanadium or molybdenum content until the summit line was reached. In contradistinction to the tensile strength, an increase in carbon content within the martensitic region leads to a decrease in creep strength according to the distance of the composition point from the summit line.

Fig. 7.—Influence of the γ-loop-forming elements upon the creep strength of steels with varying carbon content.

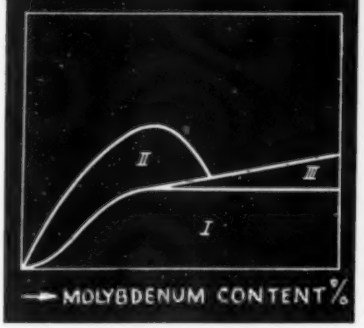


In the zone to the right of the summit line the occurrence of untransformed ferrite with increased vanadium or molybdenum content, or with decreased carbon content, causes a sharp decrease in creep strength and tensile strength. But while the tensile strength continues to decrease with increasing distance from the summit line, a continued increase in molybdenum or vanadium percentage finally leads to a minimum value of creep strength.

I.—Influence of the vanadium in the ground mass.
II.—Influence of the vanadium carbides.
III.—Influence of the solid solution Fe-V.



I.—Influence of the molybdenum in the ground mass.
II.—Influence of the molybdenum carbides.
III.—Influence of the solid solution Fe-Mo.



Figs. 8 and 9.—Diagramatic representation of the influence of the vanadium and molybdenum content upon the creep strength of heat-treated steels.

Beyond this point the direct influence of the alloying elements becomes preponderant. While in the case of vanadium steels such further increases in the vanadium percentage cause only inconsiderable increases in creep strength similar conditions cause a much larger increase in the creep strength of the molybdenum steels, this increase being accompanied by the occurrence of the Fe_3Mo_2 compound.

In both the molybdenum-vanadium and the molybdenum-vanadium-silicon steels a very rapid increase in creep strength takes place with increasing carbon content until the untransformed ferrite disappears with approximately 0·1% carbon content. In analogy to the behaviour of the vanadium and molybdenum steels of classes (1) and (2), a further increase in the carbon content of classes (3) and (4) steels, after the limit of the γ region has been crossed, is accompanied by slowly increasing creep strength with attendant increase in the martensitic portion of the structure, and a decline in creep strength is noted only with further increased carbon content.

The effect of the combined influence of the alloying elements responsible for the occurrence of the γ-loop and of the carbon percentage upon the creep strength is schematically outlined in Fig. 7. Here it is seen that for a given carbon percentage the creep strength increases with increased vanadium or molybdenum content, until with a certain content a maximum value of creep strength is reached, this maximum occurring shortly before the border of the γ-loop is reached. This is followed by a subsequent steep decrease in creep strength which mainly due to the occurrence of untransformed ferrite. A further slow increase is obtained only with very high alloy percentages. Fig. 7 shows that in correspondence with the enlargement of the γ-loop, with higher carbon content the point of maximum creep strength migrates with higher carbon contents into zones of increased alloy percentages. This graph also shows that the respective maximum values of creep strength registered for the given carbon content increase in magnitude until a carbon percentage is reached, which, in the ideal case, corresponds to the commencement of complete martensitic hardening.

It is of interest to note that the author of the original report offers two explanations of the fact that maximum creep strength values are obtained shortly before the γ-loop is reached (as evidenced by the non-coincidence of summit line and limit curve): (1) It is possible that the structure is in an unstable transient condition before the

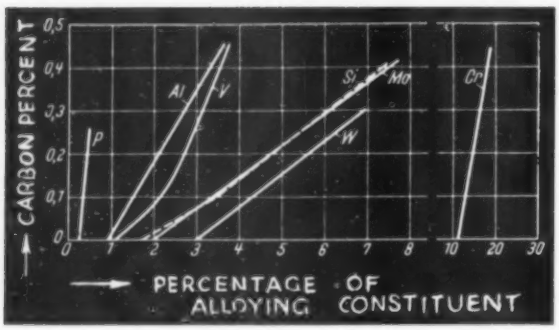


Fig. 10.—Balanced alloying element percentages required for maximum creep strength.

α-γ transformation disappears, or (2) the quenching temperature could not be raised high enough above the transformation point, as any further increase in quenching temperature would mean exceeding of the γ-loop, thus causing the occurrence of ferrite.

In order to further clarify the effect of heat-treatment upon creep strength, a comparative study was made of the test results reported by P. Gruen.[4] This comparison is said to have shown that the heat-treatment of molybdenum steels with compositions approximating to summit line values increases creep strength by 50 to 100%; while at greater distance from the summit line the influence of heat-treatment is quite inconsiderable. The same may be assumed to hold true in the case of vanadium steels.

There exist several widely different theories with regard to the underlying cause of the influence of molybdenum and vanadium upon creep strength. A. E. Wilson, C. L. Clark and R. L. Wilson[5] state that the increase in creep strength is due to the formation of the carbides of these elements. "The carbides located within the crystals, as well as the grain boundaries, exert a keying action along the crystallographic planes that decreases the tendency to creep." At the same time it is suggested by these investigators that "certain alloying elements also improve the creep resistance at a given temperature by increasing the lowest temperature of recrystallisation. Since creep and the lowest temperature, recrystallisation or equi-cohesive temperature appear to be closely connected, any element which increases this temperature will also increase the creep resistance at temperatures above it."

Holtmann believes that the influence of vanadium and molybdenum may be attributed to the three effects diagramatically indicated in Figs. 8 and 9, respectively, viz.: (i) Influence upon the ground mass by raising the temperature of recrystallisation, (ii) the action of finely distributed carbides, their action being intensified by the raising of the point of transformation, and (iii) the action of the solid solution entered into. With regard to the latter effect, it is, however admitted by Holtmann that the influence of FeV solid solutions could not be proven. It can be assumed that with high temperature and long-time tempering the influence of the carbides and of the solid solution entered into by the alloying elements with the iron becomes increasingly ineffectual because of spheroidisation. Thus there will be left only the influence (i) acting upon the ground mass. If the carbon content is too large to allow the existence of a surplus amount of vanadium or molybdenum, the influence of these elements upon the creep strength must entirely disappear with high temperature and long time of draw.

Other investigations conducted by H. Scholz and W. Holtmann[6] seem to indicate that the aforementioned characteristics of vanadium and molybdenum are shared by a considerable number of other elements showing the γ-loop. Fig. 10 shows the γ-loop limits of composition for phosphorus, aluminium, vanadium, molybdenum, silicon, tungsten and chromium as functions of carbon and alloy content. From what has been said before, it is clear enough that these limiting alloy contents would be synonymous with maximum creep strength if the discrepancy between summit line and limiting curve could be neglected.

Earlier investigations[7] point to the fact that with multiple alloyed steels the influence of γ-loop forming elements upon the structure formation can be considered as additive, but as the magnitude of influence exerted by the individual alloying element differs considerably it is difficult to postulate compositions which can be expected to show maximum creep resistance: but if it is assumed that the γ-loop forming effect of each one of several alloying elements contained in a multiple alloyed steel remains equal to that experienced with a single alloyed steel of identical carbon content, an optimum composition can be prognosticated on the basis of the following rule:—

$$\frac{(\%\ \text{Si})\ 100}{\text{Si}_c} + \frac{(\%\ \text{Mo})\ 100}{\text{Mo}_c} + \frac{(\%\ \text{V})\ 100}{\text{V}_c} + \ldots = \text{M}$$

where Si_c, Mo_c and V_c are the loop-limit percentages of the alloying constituents at the given carbon content, while M is the so-called balance factor.

It is evident that with M = 100 the steel will be nicely balanced, while M > 100 will indicate a surplus of alloying constituents, and M < 100 a corresponding deficiency. Obviously, smaller carbon contents will call for smaller percentages of alloying elements. However, care must be taken not to go below a certain carbon content, as this would lead to an unduly small critical cooling speed. A few examples of balanced steels are given in the subjoined table:—

C, %	Si, %	Mn, %	Cr, %	Mo, %	Other Elements, %	Creep Strength in Kg./sq. mm. at 500° C.	Creep Strength in Kg./sq. mm. at 550° C.
0·1	0·98	0·35	2·6	0·42	0·33 Al	—	16
0·16	0·80	0·50	1·50	1·35	0·60 V	—	35
0·20	0·95	0·44	1·66	1·67	0·65 V	55	35
0·15	0·47	0·20	2·03	1·12	0·81 V	60	45

It is claimed that this very simple rule makes it particularly easy to determine the alloy percentage required for the attainment of maximum creep strength, and that it also is very useful in determining the constitution of "substitute" steels; but it must, of course, be kept in mind that each of the alloying elements considered has its own particular effect and its individual degree of influence upon creep strength.

Control of Aluminium (No. 5) Order, 1940

Direction No. 2.

THE Minister of Aircraft Production has issued Direction No. 2 under the Control of Aluminium (No. 5) Order, 1940. This Direction came into force on the 11th of this month, and states that the maximum price of aluminium alloy scrap resulting from any machining operation, or in the form of grindings or filings, will be £50 per ton delivered free at the works of the buyer, and £44 per ton delivered free at any other place, and that for any other aluminium alloy scrap the maximum price will be £70 per ton and £65 per ton, respectively. The Direction also declares that these maximum prices will be inclusive of any packing, loading and transport undertaken by the sellers, and revokes Direction No. 1.

Copies of the new Direction may be purchased from H.M. Stationery Office, or from any book-seller. All inquiries should be addressed to the Ministry of Aircraft Production, Light Metals Control, Southam Road, Banbury, Oxon.

[4] P. Gruen. *Arch. Eisenhüttenw.*, vol. 8, 1934–35, p. 205.
[5] *Steel*, vol. 95, 1934, No. 24, p. 30; also *Oil and Gas J.*, Aug. 9, 1934.
[6] *Mitt. Kohle und Eisen Forsch.*, vol. 3, 1941, p. 47.
[7] F. Wever and A. Heinzel. *Mitt. K.W. Inst. Eisen*, vol. 13, 1931, p. 193.

METALLURGIA
THE BRITISH JOURNAL OF METALS.
INCORPORATING "THE METALLURGICAL ENGINEER"

Increased Production a Major Factor

THIS month may well prove to be of historical significance in the world conflict: during its early days a measure of retribution was meted out to Germany for the nightly avalanche of death and destruction which the people of Britain suffered at a time when there was little else to fortify their resolution than the pledge, "We shall give it to them back." The knowledge that we can now hit the enemy hard has acted like a tonic; the stubborn courage and ungrudging sacrifice of the nation, during the past two years, have earned this measure of reward, and the indications that retribution will be continued on an even larger scale have infused British people with the consciousness that slowly, perhaps, but surely, the nation is riding out the most terrible storm in its history. Disappointments and setbacks are probably still lurking around, but it is noteworthy that they are neither so shattering nor so frequent.

One of the main factors contributing to the offensive spirit which is becoming more and more manifest is the growing might of the nation, represented by the great progress made in the production of munitions of all kinds. The building up of facilities to ensure greatly increased production has involved much valuable time and an intense concentration of industry. In the early days of 1940, Britain's production of munitions was augmented by supplies of considerable volume from the United States on a cash-and-carry basis. In order to import these munitions, and other vitally important goods, Britain was obliged to maintain her exports to the highest possible level. It was for this purpose that during the early months of 1940 an export drive was promoted, and when the collapse of France made necessary further concentration upon the production of war materials, and exports had to be husbanded, the United States remained the most-favoured market, because of the need for dollar currency. Thus, in spite of the need for organising British industry for the production of munitions, to support the cash-and-carry plan, exports to the United States in the first half of 1940 were considerably greater than during the same period in 1939, and were maintained above the 1939 level during the first half of 1941.

The situation was profoundly changed in March, 1941, by the passing of the Lease-Lend Act, and subsequently by the undertaking given by the British Government, that the materials obtained under this Act, and required for the prosecution of the war effort, would not be diverted to the furtherance of private interests. This brought about restrictions and additional controls to which industry submitted with commendable cheerfulness and courage; but it was also responsible for greatly accelerated concentration on the production of munitions in this country. The results of this continuous effort are that the growing Forces are more adequately equipped for war than was thought to be possible a year ago.

While supplies of various kinds have continued to arrive from the United States, despite her own special needs as a combatant, the bulk of the needs of our Forces is being met by home industries. Mr. Lyttelton, Minister of Production, gave some idea of the enormous strides Britain has made in war production in his recent broadcast in the United States. Britain is producing tanks, "jeeps," and other mechanised vehicles at the rate of 257,000 a year; an increase of 350% over the rate in the last quarter of 1940. Big guns are being produced at the rate of 40,000 a year, and 25,000,000 rounds of ammunition are being supplied with them. Production of aircraft has increased over 100% above the rate achieved in the last quarter of 1940. Production of merchant ships has increased by 57% over the same period, even though it was thought the limit of our capacity had been reached at that time. Taking weapons of war as a whole, production has increased by 100% during the last twelve months alone.

Another factor of great significance to the successful prosecution of the war is the establishment of a combined Production and Resources Board, formed by the United States and the United Kingdom. Considerable speculation was aroused when the departure of Mr. Lyttelton for Washington became known. Now we know that for the more effective prosecution of the war Britain and the United States have decided to set up two new organisations, a combined Production and Resources Board and a combined Food Board. Thus, as regards production, there will now be a single integrated programme, adjusted to the strategic requirements of the war, while the entire food resources of Great Britain and the United States will be deemed to be in a common pool.

The Production and Resources Board will consist of the Chairman of the War Production Board representing the United States, and the Minister of Production representing the United Kingdom. It will combine production programmes of the two countries and adjust them to the needs of the Forces as indicated to the Board by the combined Chiefs of Staff. Accounts will be taken of the need to use all available production resources and to reduce shipping demands to a minimum. In collaboration with the combined Chiefs of Staff, the Board will assure continuous adjustment of the combined production programme to meet changing military conditions.

From the passing of the Lease-Lend Act onwards the separate identities of Britain and the United States have been fused in a common determination to rid the world of the present German menace and to this end Anglo-American co-operation has grown increasingly effective. The setting up of these two Boards, however, is not only an indication that for the purposes of war Britain and America are acting in complete unison, but they are of great significance to Anglo-American relations. It affords conclusive evidence of the complete confidence which each nation has in the other, and clearly indicates a solidarity of purpose.

Other significant facts, in a political sense probably as significant as the Atlantic Charter, are the agreements made between the United States and the Soviet Union and between Britain and the Soviet Union, which have just been announced as we go to Press. Many who remember the magnificent response by British industry in the "Tanks for Russia Week," which has since been maintained at a high level, will appreciate that the complete

The fact that goods made of raw materials in short supply owing to war conditions are advertised in "Metallurgia" should not be taken as an indication that they are necessarily available for export.

understanding, which has now been reac hed between the two countries, will have a great influence on the production of munitions in this country, which is bound to have very severe repercussions on Germany.

Co-operation between the United Nations is progressing at a rapid rate, many difficulties have been overcome and others, no doubt, remain to be solved before the world can again move forward along the broad highway of civilisation, but there must be no slackening of effort, the men and women of offices and workshops have the same responsibilities as those in the various armed forces, and it is for each individual to ac.ept his or her responsibilities and discharge them in the most competent and useful way. Much of an outstanding character has happened this month, and a feeling of optimism is prevalent; but there is still a hard road to traverse which can only be accomplished by each giving his or her best to the war effort, whatever the position of the individual.

Metal Rectifiers Applied to Electrodeposition

THE well-known copper-oxide rectifier, of which a capacity of over a million amperes has been supplied to the electrodeposition industry, was described by D. Ashby at a recent meeting of the Electro-depositors' Technical Society, an improvement as a result of many years' research having been found in the selenium rectifier, which may be operated under oil where it is immune from attack by the atmosphere of the plating-shop and is effectively cooled. The rectifier is mounted in a tank, together with the transformer, if space permits, a number of tubes being fitted to the tank sides to increase the cooling surface. This is found to be superior to the use of water cooling, which is dependent on the continuity of the supply of water, for which an alarm system should be included to protect the rectifier should the water supply be reduced. This problem does not arise with natural cooled equipment.

A three-phase, half-wave selenium rectifier is used for outputs of 8 volts and less, which enables an overall efficiency of 75–80% to be obtained, which does not drop below that value until the current is reduced to a quarter of the normal rating. Opinion has been expressed that the ripple in this circuit would not permit satisfactory plating, but practice has proved that there is no justification in this argument, results on all solutions and processes having been found identical with those of a generator. A single phase rectifier, however, is not recommended for chromium plating, but a three-phase plant may be operated with every satisfaction from a single-phase power supply by means of a static-phase converter.

A method of protecting the rectifier against overloads and of giving an alarm should the current increase or decrease beyond certain limits has been developed; this is of particular interest where the deposition process is lengthy and conditions in the bath may vary so the current does not remain at the initial value.

Another recent development is the use of a half-wave rectifier and stand-by battery, which provides a polarising current to the bath should the power supply fail to the plating rectifier. The stand-by battery is permanently connected to the bath and is usually trickle charged from the main rectifier by the leakage current through the half-wave rectifier, through which it can discharge freely should the main transformer become dead, and so continue to supply the bath at a reduced rate. This scheme is of particular importance in nickel deposition when the work may rapidly be spoilt if the current is interrupted.

Selenium rectifiers are also used for operating variable speed D.C. motors up to 10 h.p. capacity on automatic plants, etc.

Forthcoming Meetings

Iron and Steel Institute

Joint Meeting in Sheffield

A Joint Meeting, organised by the Iron and Steel Institute, in conjunction with the Sheffield Society of Engineers and Metallurgists, the Sheffield Metallurgical Association and the South Yorkshire Section of the Institute of Chemistry, will be held in the Assembly Room, Royal Victoria Station Hotel, Sheffield, on Wednesday, June 24, 1942, at 7 p.m. Dr. W. H. Hatfield, F.R.S. (Vice-President of the Iron and Steel Institute, President of the Sheffield Society of Engineers and Metallurgists), will take the chair.

The following papers will be presented for discussion :—"First Report of the Standard Methods of Analysis Sub-Committee," a Sub-Committee of the Committee on the Heterogeneity of Steel Ingots (a Joint Committee of the Iron and Steel Institute and the British Iron and Steel Federation, reporting to the Iron and Steel Industrial Research Council). To be presented by Dr. E. Gregory, Chairman of the Sub-Committee. "On the Carbide and Nitride Particles in Titanium Steels," by William Hume-Rothery, M.A. (Oxon), D.Sc. (Oxon), F.R.S., Geoffrey Vincent Raynor, M.A. (Oxon), Ph. D.(Oxon), and Alexander Torrance Little, D.Sc. (Glasgow), (Inorganic Chemistry Laboratory, University Museum, Oxford). To be presented by one of the authors.

Institute of British Foundrymen

Readers are reminded that the Thirty-ninth Annual General Meeting of the above Institute will be held at the Waldorf Hotel, Aldwych, W.C. 2, on June 20, 1942. After the normal business meetings a number of technical papers will be presented for discussion, particulars of which were given in our last issue.

Electrolytic Polishing of Silver*

SILVER can be polished electrolytically by a process of anodic corrosion in a bath of silver cyanide, potassium cyanide and potassium carbonate such as is usually employed for silver plating. Careful regulation of current density is required. Room temperature is most suitable for the operation, although slightly higher or lower temperatures are equally satisfactory. Sufficient stirring to prevent layer formation is desirable, but high stirring rates are to be avoided. The reflectivity of the resulting surface appears to be as high as, if not higher than, that of a mechanically poilished surface. The process depends upon the formatiom of an anode film which is soluble in the electrolyte under suitable conditions of current density, temperature, and free cyanide concentration.

Electropolishing affords a means whereby silver specimens may be prepared for etching and microscope examination without introducing any possibliity of the production of a mechanically work-altered surface layer. After polishing the etching may be carried out in one of the usual ways, that is, by the use of a corrosive reagent such as chromic acid or by making the specimen anode in a cyanide plating bath and etching at a low current density (0.4 amp./dm.$_2$ or less).

Silicon Bronze for Sand Castings

IN our last issue, under the above heading, we mentioned that arrangements were being made to produce and market the special alloy hardener used in the making of P.M.G. metal. We have since been advised that Messrs. A. Cohen and Co., Ltd., 148 and 149, Great Dover Street, London, S.E. 1, have taken over the manufacture of this special hardener and also ingots. This hardener can be used as a substitute for tin in the compounding of many non-ferrus alloys.

* L. I. Gilbertson and O. M. Fortner, The Electro-chemical Society, General Meeting, April, 1942.

Chilled Wheels for Streetcars

By William F. Chubb, Ph.D., B.Sc.

There are considerable differences in the practices employed for the production of wheels for rail transport vehicles. In this article a brief description is given of the production of solid-cast wheels for freightcars and street railcars. Attention is directed more especially to certain researches undertaken to improve the quality of the product and its life in service.

IN the production of wheels for freightcars and street railways, practice in Europe differs very considerably from that in the United States and Canada. On the eastern side of the Atlantic a preference has developed for tyres of forged steel shrunk on to the centre, whereas in American practice solid-cast wheels are frequently specified, more particularly as regards wheels for streetcars. For this reason there has been built up a fairly large industry for the casting of these wheels, and it is the purpose of this article to give an account of certain researches undertaken in attempts to improve the quality of the product and the life in service of these materials. Before passing to details of this research it might be as well, however, to provide a brief general account of the methods of production with which it was associated.

Production Methods

The general procedure for manufacturing such wheels consists in the production of a white iron to meet certain fairly well-defined properties. The essential demands are a sound material of grey structure with a hard tread and flange, produced by suitable chilling, to resist wear. In addition, a certain degree of toughness is essential to withstand the shock inevitably imposed at catchpoints on the tracks. To provide sufficient life in service a fairly considerable depth of chill is required, and the provision of a deep chill has the advantage that when wear results the wheels may be trued up again for further service. It will also be clear that the conditions of service stipulate for a fairly progressive change of structure from the hard-wearing chilled surfaces of the flange and tread to the softer grey interior structure. In production these desired properties are provided by chill casting the tread and the flange, and in this way a white structure is obtained to a depth of, approximately, $\frac{11}{16}$ in. to $\frac{13}{16}$ in. before the appearance of the intermediate mottled section. These irons are cupola melted, and wheels of reasonable service performance have been successfully produced for many years past without additions of special alloying elements. Of recent years, however, there has been a tendency to increase the loadings of freightcars, and streetcars have been speeded up to satisfy modern traffic conditions, and these changes have resulted not infrequently in service troubles, due to excessive wear on the treads and flanges of the wheels. As will be clear, these new conditions of service may also impose an excessive thermal shock during braking, and a demand has accordingly arisen for a new type of iron to meet these changed conditions.

In the production of these wheels, difficulties may sometimes arise due to the formation of internal cracks. As an inevitable consequence, it is common experience that these cracks do not always reveal themselves as definite flaws until some time after the wheels have been placed into service, when a claim for free replacement may arise. Other difficulties in production are concerned with choice of suitable heat-treatment. Such heat-treatments generally have a twofold object, namely, stress-relieving after casting and the production under controlled conditions of a certain desired type of structure. To achieve this, the practice most frequently adopted is to remove the wheels from their moulds within a predetermined time after pouring, and to pile them in pit-type furnaces previously heated to such a temperature as to provide the desired rate of cooling. The wheels are then transferred from this primary pit after being allowed to cool to a temperature of about 850° F., and they may then be given a further and similar treatment in a second pit. As an alternative procedure, however, the castings are sometimes stacked, after treatment in the primary pit, in a well-lagged chamber and allowed to cool to ordinary temperatures. Yet a third treatment which has been known to be successful is that of stacking the hot castings in this chamber and cooling slowly to ordinary temperatures. Owing to the many treatments that may be given, there is indeed a conflict of opinion as to the most appropriate cycle of heat-treatment necessary to convey to the iron the desired structural and physical characteristics.

In testing wheels after heat-treatment, a selected wheel is usually broken up and the load necessary to fracture is recorded. Any unusual behaviour is noted, and a transverse section through the tread and flange is prepared by cutting with an elastic wheel.

Service conditions demand a minimum depth of chill of

Fig. 1.—Standard wheel, tread, chilled edge, × 50, unetched.

Fig. 2.—Standard wheel, tread, mottled portion, × 50, unetched.

Fig. 3.—Standard wheel, grey section, × 50, unetched.

Fig. 4.—Standard wheel, tread, chilled edge × 50, etched with 4% picric acid in alcohol.

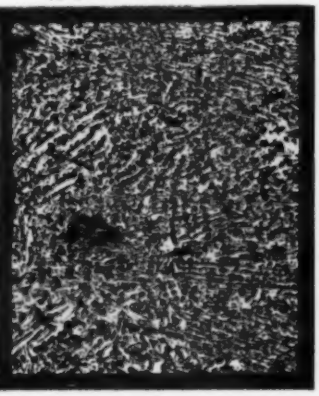


Fig. 5.—Standard wheel, tread, mottled portion, × 100, etched with 5% nitric acid in alcohol.

Fig. 6.—Standard wheel, grey section, × 200, etched with 5% nitric acid in alcohol.

Fig. 7.—Standard wheel, tread, chilled section, × 500, etched with 5% nitric acid in alcohol.

Fig. 8.—Standard wheel, flange, chilled edge, × 50, unetched.

the order previously stated, and appropriate readings of hardness are accordingly made by means of the scleroscope at succeeding depths of $\frac{1}{16}$ in. below the tread surface. At the tread and flange surfaces the required hardness is approximately 66 scleroscope, and Brinell hardness at these surfaces should be about 460 in unalloyed chill-cast irons.

Standard Wheel

For a typical analysis of iron employed in the production of chill-cast wheels, the following may be quoted :—

	%		%
Total carbon	3·44	Sulphur	0·130
Combined carbon	0·79	Phosphorus	0·278
Graphite carbon	2·65	Silicon	0·60
Manganese	0·68	Nickel	Nil
		Chromium	Nil

The established practice at one works specialising in the production of these wheels is to pour at about 2,500° F. and to transfer to the pit furnace after 30 mins. in the mould, the pit being preheated to 1,220° F. After sealing the pit and cooling over a period of about 44 hours the wheels are removed at a temperature of approximately 850° F.

The microstructure of such a wheel will be understood by reference to the accompanying illustrations. Fig. 1 depicts the extreme edge of the tread in an unetched condition. As will be evident, the material is practically free from graphite to a depth of approximately 0·005 in. At this depth, however, small nests of graphite make their appearance and the iron then contains also numerous pin-points of graphite, which grow progressively in both number and size at increasing depths below the tread surface. The mottled section of the wheel is then met, as illustrated in Fig. 2, and here it will be seen that the graphite takes the form of rosettes or clusters in a ground mass containing finely dispersed specks of graphite. In the central and unchilled section of the wheel the structure is that shown in Fig. 3, unetched at a magnification of 50, the graphite here being present in the form of rather coarse flakes.

In the etched condition the area illustrated in Fig. 1 has the structure shown in Fig. 4, the magnification of the two photomicrographs being 50. This structure, developed by 4% picric acid in alcohol, consists of primaustenoid (white) and pearlite (dark), with two black areas of graphite, as in the unetched photomicrograph of Fig. 1. As will be clear, this structure is of an inerlocking type demanded by service requirements, and there is little or no evidence of columnar growth in the primaustenoid. For a typical etched structure of the mottled section, Fig. 5 should be studied, and here it will be apparent that the dark graphite flakes cannot be too readily distinguished, the reason being that they are intimately associated with the dark-etching pearlite. The grey portion of this wheel, after etching with 5% nitric acid in alcohol, as in Fig. 6, shows more clearly at a higher magnification than in the unetched photomicrograph of Fig. 3 the relative coarseness of the graphite flakes in this section.

A particularly interesting photomicrograph is that of Fig. 7, illustrating the character of the pearlite areas in the chilled portion of the wheel, close to the tread surface. This constituent is, as shown, essentially lamællar in structure, but there is revealed at the high magnification noted a certain degree of spheroidisation, resulting during the heat-treatment described above.

Fig. 9.—Standard wheel, flange, mottled section, × 50, unetched.

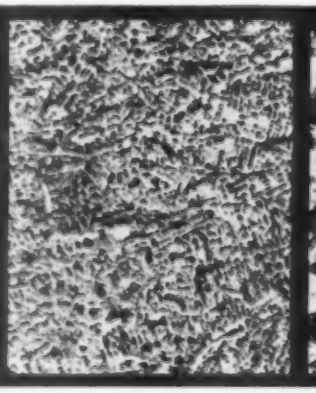


Fig. 10.—Standard wheel, flange, chilled edge, × 50, etched with 4% picric acid in alcohol.

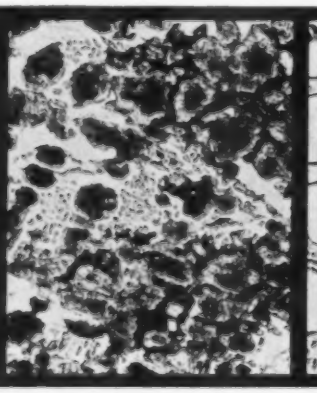


Fig. 11.—Standard wheel, flange, mottled section, × 100 etched with 5% nitric acid in alcohol.

Fig. 12.—Standard wheel, flange, chilled edge, × 500, etched with 5% nitric acid in alcohol.

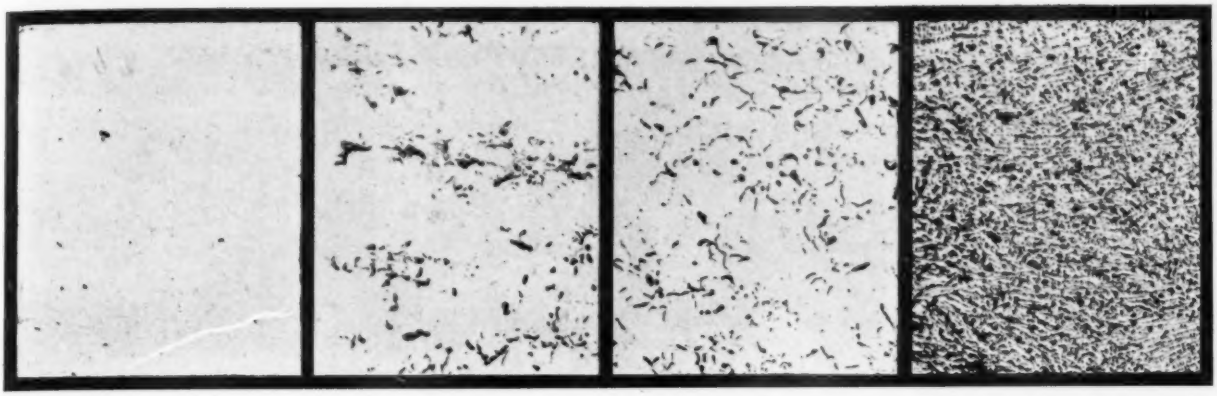


Fig. 13.—Alloy iron, tread, chilled edge, × 50, unetched.

Fig. 14.—Alloy iron, tread, mottled section, × 50, unetched.

Fig. 15.—Alloy iron, grey section, × 50, unetched.

Fig. 16.—Alloy iron, tread, chilled edge, × 50, etched with 4% picric acid in alcohol.

While all the structures described above concern the tread of a standard chilled wheel, the structure of the flange may now be considered. Service requirements again demand a hard-wearing material, the structure in the present instance being that illustrated in Fig. 8. It will be observed that graphite flakes exist immediately below the surface, as in the tread section of Fig. 1. In the mottled areas the graphite flakes of Fig. 9 are essentially of the same type as those of the tread section illustrated in Fig. 2. There is often, however, a greater tendency to form rosettes or clusters in the flange than in the chilled tread.

In the corresponding etched sections of the wheel flange an interlocking type of structure of primaustenoid (white) and pearlite (dark) is again to be seen, as in Fig. 10, but by comparison with the tread structure this photomicrograph reveals a greater degree of columnar growth in this section of the casting. As might be expected, the structure of the mottled section under the flange, as depicted in Fig. 11, is essentially similar to that under the tread of the wheel. Finally, the characteristics of the pearlite areas are clearly illustrated in Fig. 12, this constituent being in a well-defined lamellar form.

Nickel-Chrome Wheel

On the basis of the above investigations an attempt has been made to produce a wheel of denser structure and superior strength properties, while retaining at the same time the required type of interlocking structure at the wearing surface, as described previously. For this purpose a special iron alloyed with nickel and chromium was made and cast into a wheel of standard dimensions under conditions almost identical with those prevailing in the founding of the standard unalloyed wheel. As this is possibly the first instance recorded of a streetcar wheel being produced in nickel-chrome iron, the analysis may be quoted as below :—

	%		%
Total carbon	3·28	Sulphur	0·10
Combined carbon	0·83	Phosphorus	0·158
Graphitic carbon	2·45	Silicon	1·00
Manganese	0·49	Nickel	1·76
		Chromium	0·47

It should be noted that to counteract the increased hardness induced by the two alloying elements, the silicon was here raised from 0·60% to 1·00%. A base iron of lower phosphorus content was, however, employed in this instance, and the casting was subjected to a heat-treatment exactly similar to that given to the unalloyed iron previously described.

A comparison of structures in corresponding sections of the two wheels is highly instructive, but before passing to a more detailed discussion of this it may be as well to record that in so far as hardness is concerned the alloyed iron is of lower hardness at the chilled surface than the unalloyed material. Thus, the unalloyed chilled iron gave a scleroscope reading of 66 and a Brinell hardness of 460; while the flange of the nickel-chrome wheel was of 56 scleroscope and 390 Brinell, and the tread of 61 scleroscope and 430 Brinell.

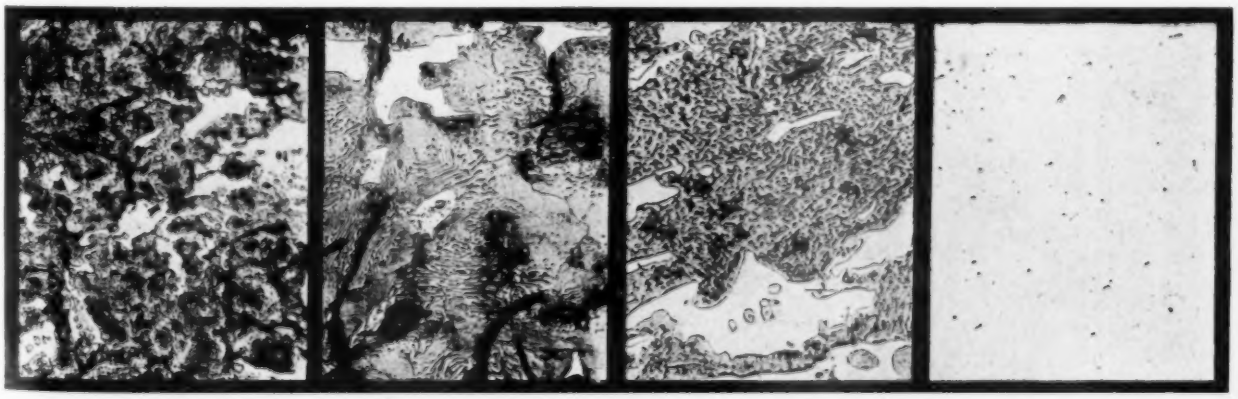
In Figs. 13 to 24 are provided illustrations of the structures obtained in this nickel-chrome wheel. Of these, the first depicts the unetched material immediately below the tread surface. By contrast to the unalloyed wheel shown in Fig. 1, this section contains no coarse graphite, but in the mottled section beneath the chilled portion of this wheel the graphite will be seen to exist in a form very different from that in the corresponding section of the standard wheel. As revealed in a comparison between Figs. 14 and 2, the graphite flakes of the alloyed iron are very much finer.

Fig. 17.—Alloy iron, tread, mottled section, × 500, etched with 5% nitric acid in alcohol.

Fig. 18.—Alloy iron, grey section, × 500, etched with 5% nitric acid in alcohol.

Fig. 19.—Alloy iron, tread chilled edge, × 500, etched with 5% nitric acid in alcohol.

Fig. 20.—Alloy iron, flange, chilled edge, × 50, unetched.



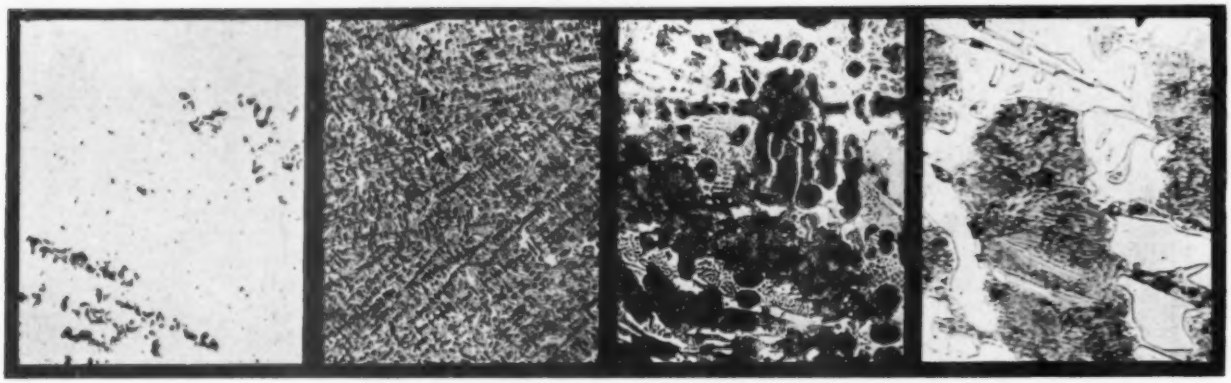


Fig. 21.—Alloy iron, flange, mottled section, × 50, unetched.

Fig. 22.—Alloy iron, flange, chilled edge, × 50, etched with 4% picric acid in alcohol.

Fig. 23.—Alloy iron, flange-mottled section, × 100, etched with 5% nitric acid in alcohol.

Fig. 24.—Alloy iron, flange, chilled edge, × 500, etched with 5% nitric acid in alcohol.

For the grey portion of this wheel Fig. 15 provides a representative structure, and by comparison with the structure of the unalloyed iron, as provided in Fig. 3, it is again clear that the graphite has formed into much finer flakes.

In the etched condition the structure of the chilled portion immediately below the tread surface, as in Fig. 16, is apparently much more dense than that of the standard wheel shown in Fig. 4. To illustrate the structure of the mottled portion, Fig. 17 should be examined in conjunction with Fig. 5, when it will be seen that the structure of the nickel-chrome iron is much more dispersed than in the standard unalloyed iron.

A comparison of structures after etching is also of considerable interest in revealing that for the grey section of the alloy iron, Fig. 18, the graphite flakes are finer and the pearlite constituent more definite in structure than in the unalloyed iron of Fig. 6. Finally, to complete this study of microstructures in the tread section, Fig. 19 illustrates at a magnification of 500 the pearlitic areas of the chilled portion. Here it is worthy of note that the structure is very different from that of the pearlite in corresponding sections of the standard wheel, for, by examining Figs. 19 and 7, it will be evident that very little of the pearlite in the standard wheel has been spheroidised during the heat-treatment, whereas in this nickel-chrome alloy there exists a preponderating amount of spheroidised cementite. This type of structure is naturally indicative of a much stronger and tougher material, though, as already mentioned, the tread and flange are of lower hardness than those of the standard iron.

As far as the flange of this nickel-chrome iron is concerned, the unetched structure is that shown in Fig. 20. Here will be seen a small amount of very fine graphite, well distributed throughout the section, whereas in the corresponding structure under the tread this alloyed material contains little or no such graphite. In the more mottled sections of this wheel the form of the graphite, illustrated in Fig. 21, is again very different from that of the corresponding section in the standard wheel of Fig. 9, there being an almost complete absence of graphite in rosette form.

In the etched condition, the chilled section of this flange will be seen in Fig. 22 to consist of interlocking dendrites of primaustenoid (white) and pearlite (dark), as in the unalloyed iron of Fig. 10, but it will be observed that the pearlite assumes a more dendritic form than the pearlite of the corresponding section of the standard wheel.

The fact that this nickel-chrome iron is stronger and tougher than the standard wheel is again confirmed by a comparison of structures in the mottled sections of the two flanges. As illustrated in Fig. 23, the general structure is very similar to that of Fig. 11, but it should be noted that for the nickel-chrome wheel the pearlite constituent is less well defined or more sorbitic than the pearlite in the mottled section of the unalloyed iron. Finally, this somewhat indefinite and less pearlitic structure in the nickel-chrome wheel is very well illustrated in Fig. 24, and the differences of structure here recorded are very clearly revealed by comparison with Fig. 12, which depicts the structure of the standard wheel at the same magnification.

A summary of the results of the investigation and the author's conclusions will be published in the Juy issue.

High Temperature Lubrication

THE lubrication of the bearings, chains and other moving parts of the various types of annealing and heat-treatment furnaces has always been a problem. The many different types of furnaces now employed equipped with mechanism for loading and discharging, such as pushers, walking beams, mesh belts, pan conveyers, rotary drums, continuous strip, rotary hearths, and many other types, operate under varying degrees of temperature which are frequently high, and it has been the practice to use a heavy oil or grease with a suitable filler. A difficulty encountered is the amount of carbon formed, due to the cracking of the oil or grease. This accumulates and interferes with the function of the working surfaces involved. Another factor is the possibility of smoking of the hydrocarbon contaminating the atmosphere in the furnace chamber.

Many of the difficulties encountered with heavy oil or grease lubricants at high temperatures have been overcome by the use of "dag" colloidal graphite in a light mineral oil carrier which evaporates to leave little or no carbon residue. An advantage of light oil is that it volatilises speedily without undue smoking and is mostly removed before the part to which the lubricant has been applied enters the furnace zone. The graphited surface formed by this lubricant provides adequate lubrication up to temperatures well beyond the boiling point of oils and greases.

The best method of treating oven and furnace chains or the bearings of furnace cars and the like, is to spray on or apply the graphited lubricant when the parts requiring treatment emerge from the hot zone. The residual heat then drives off the carrier, so that when the part re-enters the furnace only a slippery film of graphite remains. Where automatic or drip-feed lubricators are being employed one of several graphited lubricants can be employed in the normal manner. Further information on these lubricants may be obtained from the Technical Department of E. G. Acheson, Ltd., 9, Gayfere Street, London, S.W. 1.

The Cold-Pressing Properties of Duralumin-Type Alloy Sheets,

with Special Reference to the Production of Large Pressings for Aircraft

Difficulties encountered in the cold-pressing of Duralumin-type alloys and methods used in overcoming them are discussed. Investigations have been made with a view to correlating the mechanical properties, microstructure and deep drawing properties of Duralumin to B.S.S. 5L3, and it is shown that improved drawing properties may be obtained by modifying the process of manufacture.

IN order to facilitate and increase speed in the production of aircraft, die-pressing was adopted for the outer panelling and airframe parts, formerly produced by hand, and many difficulties were encountered, since the material used for the panels had pressing properties considerably inferior to those on which press-shop managements and personnels had built up their experience. On account of their high strength-to-weight ratios, Duralumin-type alloys were in general use for these parts, and as there were very few data available on the cold pressing of these alloys, investigations, described by J. C. Arrowsmith, K. J. B. Wolfe, and G. Murray,* were undertaken with a view to assisting press-shop production.

Since the corrosion resistance of this type of alloy is considerably lower than that of pure aluminium, the alloys are mostly available in the aluminium coated or " Alclad " form. Due to the fact that the aluminium coating is extremely soft and scores very easily, the coated alloys are more troublesome in the press-shop than those uncoated. The aluminium coating of these alloys normally amounts to 10% of the total thickness, and the low strength of the coating reduces the overall strength of the material used for the core. To compensate for this, and to provide the designer with an aluminium coated material equal in strength to an uncoated sheet, aluminium alloy sheet to D.T.D. Specification 390 employs a core alloy of higher strength. The improvement in tensile properties, however, is obtained at some sacrifice of ductility, and the cold-pressing properties of this alloy are inferior to those of Duralumin to B.S.S. 5L3.

Pressing difficulties common to both coated and uncoated alloys fall into two categories :—

(*a*) Those associated with the fundamental properties of the alloys ; and

(*b*) Those caused by defects in the alloys.

Under the term " fundamental properties " of the alloys, the authors discuss (1) effect of heat-treatment ; (2) low ductility compared to other deep-drawing materials ; and (3) high " spring-back " compared with other deep-drawing materials.

Effect of Heat-Treatment

The Duralumin type alloys develop their strength as a result of solution heat-treatment, which consists of a water quench from a temperature in the neighbourhood of 500° C., followed by an age-hardening process at room or slightly elevated temperatures. Immediately after quenching, the alloys are soft and comparatively ductile, and are amenable to cold-pressing. The alloys which age-harden at normal temperature are usually received from the supplier in the solution heat-treated and fully aged condition, and it is the authors' experience that even for easy shapes it is advisable to solution heat-treat the blanks and to carry out the forming operation immediately afterwards. Unless the blanks are re-solution heat-treated, trouble is invariably experienced with tearing at the edges, when work-hardening has resulted from the shearing or blanking operation.

Buckling is one of the first difficulties encountered when blanks are quenched in water after solution heat-treatment in a salt-bath. A considerable degree of distortion results from the quenching of large blanks which subsequently require roller levelling, as a badly distorted blank will result in a buckled pressing unless the amount of stretching in the forming die is sufficient to overcome the difficulty. Removing the buckles from blanks in a levelling machine has the disadvantage of work-hardening the material, which impairs its cold-pressing properties and increases the tendency to spring back, but the work-hardening by this method is not so pronounced as by stretching or cold-rolling methods.

Distortion on quenching results from differences in the cooling rate ; rapid immersion by effecting a more uniform rate of quenching minimises buckling to some extent, but while this is frequently possible with small parts, uniform quenching of large parts is not easy. Buckling can also be reduced by delaying quenching of the solution heat-treated blank ; experiments have shown that though this practice lowers the mechanical properties obtained after ageing, it is possible to achieve a useful reduction in the amount of buckling with lowering the mechanical properties below specification requirements ; delayed quenching, however, is not considered to be advisable. Oil-quenching will reduce buckling, but is not satisfactory when the material is solution heat-treated in a salt-bath. Quenching in hot water (80°–100° C.) was found to reduce buckling, but a certain amount of ageing occurred at the elevated temperatures, which gave the material less satisfactory drawing properties. Fog or spray quenching, if properly applied, reduces distortion and buckling, but the chief practical difficulty is the production of a complete and instantaneous blanket of spray over a part of large surface area.

With regard to artificial age-hardening alloys which may be age-hardened after pressing, the authors point out that unless the thickness of metal is considerable, above 14 S.W.G. (0·080 in.), there is a serious risk of distortion occurring during the process of age-hardening at elevated temperatures and as a result the finished pressings may require adjustment of shape when they are in their hardest condition.

The authors consider the refrigeration storage of solution heat-treated Duralumin at — 7° C., at which temperature blanks may be kept successfully for four days. Refrigerated trucks may be used to convey solution heat-treated blanks from heat-treatment to the press-shop, but they draw attention to the fact that the low ductility of Duralumin-type alloys often requires pressings to be produced in several operations, with intermediate heat-treatment to restore ductility. Solution heat-treatment followed by quenching is used for this purpose, but it may cause distortion, and grain growth may occur in those parts of the pressing which have been critically strained.

When working with blanks which had been stored for short periods in a refrigerator, it was noticed that they appeared to have improved pressing properties. This was investigated, using tensile test-pieces of 5L3 heat-treated

* *Jour. Inst. Metals*, April, 1942, 109–132.

and quenched and then stored at — 18° C., — 10° C., and at room temperature, tensile tests being made after varying periods of storage. The results show that the maximum ductility as indicated by the percentage elongation of solution heat-treated Duralumin alloys is not obtained immediately after the water quench. The ductility appears to rise to a maximum after an interval of time which is dependent upon the temperature of storage, and then to fall continuously as age-hardening proceeds. At room temperature this interval is of the order of 10 mins., and is, therefore, too short to be put to any practical use. At normal refrigerator temperatures (— 6° to — 10° C.), however, the interval is approximately 2 hours, while at — 18° C., the maximum value for percentage elongation is obtained 4 hours after quenching. Repeated tests showed that the normal variations in elongation are much less than the changes in properties under investigation.

The authors have found in practice that material which has been stored for 2 to 4 hours in the refrigerator will frequently give satisfactory pressings under the same die conditions as those that are giving split pressings on blanks taken straight from the quenching operation. It has been found advantageous on any blanks that give trouble immediately after quenching. Investigations on the effect of time at temperature of solution heat-treatment showed that no useful purpose was served by prolonging the time beyond 40 mins.

Spring-Back Effect

During the production of any pressing, the maximum deformation of the material occurs when the punch reaches the bottom of its stroke. When the punch starts to rise there is always a certain amount of elastic recovery in the material, which is popularly termed "spring-back." The extent of this elastic recovery depends on a number of factors, including Young's modulus, the nature of the forming stresses—e.g., bending, tension, and compression—spring-back being greater where the metal has been bent to shape than where it has been stretched,—and the value for the elastic limit of the material. Spring-back is further affected by the work-hardening, which changes the elastic limit of the material, and by the drawing conditions. As a result, it has been found impossible to calculate the correction necessary for the punch, and it has been necessary to make adjustments to the punch shape during the try-out stage of the die manufacture. More recently, spring-back difficulties have been successfully overcome by a method in which the pressing is taken almost to full depth under conditions of easy flow of metal through the blank holders, after which blank holder pressure is adjusted to prevent any further slip, and the punch then taken to the bottom of the stroke. In this manner the amount of stretching necessary to minimise spring-back is obtained, together with the easy flow through the blank holders in the initial stages of the draw, which is necessary to prevent splitting the metal.

As regards difficulties due to defects in Duralumin-type alloys, scratches and other surface defects have frequently been traced as a source of splitting troubles during the forming operations. It is, therefore, essential to handle all blanks, and Alclad blanks in particular, with great care during the heat-treatment operations. It has been found also to be an advantage to have the edges of the quenching and washing tanks lined with wood to eliminate danger of surface scoring.

In an attempt to correlate physical properties and the deep-drawing properties of Duralumin 5L3, samples taken during press runs, when all or none of the pressings were splitting, were tested. The elongation of test-pieces was measured in directions both parallel to and at 90° to the final mill rolling, and micro-structure was examined, particularly in the longitudinal direction.

8 in. gauge test-pieces were used, the elongation being noted on 8 in., together with the elongation on the 2 in., which included the fracture. The 0·1 and 0·5% proof stresses were also determined. Plottings of the various results obtained indicate that material having elongations on 8 in. and 2 in. greater than 17·5 and 22·5% respectively in conjunction with maximum values of 8·5 tons/sq. in., for 0·1% proof stress and 20·3 tons/sq. in., for maximum stress will probably show satisfactory pressing results.

Unsatisfactory pressing results were associated with a microstructure in which the intermetallic compounds were in the form of long strings. On the other hand, material which possessed good pressing properties showed the intermetallic compounds to be broken up and more uniformly disseminated throughout the matrix.

Following the observations on microstructure as it affected drawing properties, suggestions were made to suppliers of Duralumin sheet to modify their rolling technique in a deliberate attempt to produce material having the desired type of microstructure. This involved an increase in the amount of cross-rolling in the hot mill, and also a decrease in the amount of reduction in the cold mill. Sample lots of sheets produced in this way have been tried on difficult pressing, and their improved drawing properties have been clearly demonstrated.

Deep-drawing properties of various aluminium alloys have been compared in an experimental double-action die producing an irregular dish-shaped pressing from a blank 13½ in. in diameter. Wrinkle-free pressings of increasing depth were produced by adjustments of blank holder pressure and the stroke of the punch until splitting of the metal took place during pressing. The depth in notches of the deepest pressing which could be consistently produced without splitting was taken as the measure of the deep-drawing properties of the alloy being tested. From the results, it would appear that the special type of Duralumin in the solution heat-treated condition has drawing properties approaching those of ordinary Duralumin in the fully annealed condition.

The Effect of Hydrochloric Acid Plus Inhibitor on the Corrosion Resistance of 18-8 Stainless Steel

IT is common practice to improve the corrosion resistance of 18-8 stainless steels by so-called "passivation" in nitric acid. The improvement is not unique to oxidising acids, because it is shown by H. H. Uhlig and E. M. Wallace* that surface treatment with hydrochloric acid, 3 N to 12·5 N, containing an inhibitor, accomplishes much the same thing.

A commercial pickling inhibitor and quinolin ethiodide were the inhibitors tried. Corrosion resistance was determined by exposure to 10% ferric chloride, noting weight loss and number of pits. The number of pits per specimen approximately paralleled the weight loss.

Improvement in corrosion resistance was better for specimens surface-treated with the higher concentrations of acids, except 12·5 N hydrochloric acid. The latter produced the same or lower resistance to pitting as compared with 9 N acid. Improvement upon using hydrochloric acid plus quinolin ethiodide was less, but comparable with the improvement by nitric acid.

It appears plausible that "passivating agents" for 18-8 including nitric acid improve 18-8 not by increasing the thickness of a supposed protective oxide film, but by preferential solution of anodic areas, thereby forming a metal surface electrochemically homogeneous and free from pit nuclei. This preferential solution of surface metal is accompanied by mild etching which smooths and brightens, hence improves the appearance. Oxidising acids and anodic treatment in addition to equipotentialising the surface remove interstitial hydrogen, which when present may be a barrier to maximum passivity of the alloy.

Electric furnaces and muffles for laboratory use are discussed in a folder recently issued by Johnson, Matthey & Co. Ltd., Hatton Gardens, London, E.C.1.

* The Electrochemical Society. Preprint, 81-25.

"Dead" Steel

By Bernard Thomas, F.Inst.P., F.Inst.F.

Many irregularities encountered in heat-treatment are due to a lack of uniformity in the heating of ingots or billets to be rolled or forged; a steel that would normally respond readily to heat-treatment operations may show lack of "life" from this cause; the author cites such an instance.

OF "killed" steel we know, but "dead" steel may be an expression new to many readers.

It was in the experience of the writer in the heat-treatment of certain steels which conformed approximately to the upper limits of the old specification B.S.S. 5005/203, but where hardness was desired of the order of 500/550 Brinell, that some casts failed completely to respond to hardening treatment. Readings of merely 250/300 Brinell would be forthcoming on quenching, which were quite useless for the purpose involved. Invariably in such cases the analysis would conform satisfactorily to requirements, but fractures were so dull and lifeless as to have bestowed upon them the soubriquet "dead" steel.

Typical analyses of two such steels are given in Table I, and it will be seen that no exception could be taken of either.

TABLE I.
TYPICAL COMPOSITION OF TWO "DEAD" STEELS.

	A		B
	%		%
Carbon	0·51	..	0·50
Manganese	0·79	..	0·75
Sulphur	0·029	..	0·041
Phosphorus	0·055	..	0·045
Silicon	0·137	..	0·121

The tools for treatment were forged out into plates measuring roughly 11 in. long × 7 in. wide × 0·130 in. in thickness, and hardening was carried out normally by quenching in fused salts from 800°–820° C., according to the actual analysis, and followed by a suitable temper to yield the desired Brinell readings. The latter temperature was likely to vary from 240°–300° C. The employment of fused salt as the quenchant may be qualified by the finer structure of martensite so obtained, in comparison with the oil used as a medium, and which constituent in such condition lends itself much more readily to the development of the next in the stage of transition—namely, troostite—in a state of distribution which yields higher impact values for given tensile strengths.

In dealing with work of this character, one becomes accustomed to the usual hardening difficulties, such as heavy oxide or layers of scale acting as an insulator of some degree which prevents a thorough quench. Or, again, surface decarburisation due to the heating for forging. Sulphide and/or phosphide segregation yielding areas dissimilar in character to the main body of the forging, low carbon or low manganese, or even high sulphur combining with the latter to reduce its influence, are all fairly well-known trials and tribulations, but the "dead" steel appeared to be a new phenomenon.

Usually in such cases, physical defects absent, chemical analysis becomes the next resort. Unfortunately, as explained previously, the full analysis conformed to specification and there appeared no reason for hardening to fail.

In view of the lifeless appearance of the fracture, a micro-examination was made, but here again a blank was drawn, as there appeared no indication of either burning or overheating, neither was there any trace of undue oxidation or segregation.

As no method of examination that could be conceived seemed able to offer solution, attention was turned to manufacturing particulars at the steelmakers, which latter offered every facility for so doing.

Both charges and additions, melting times and temperatures, etc., etc., were of a monotonous similarity to a discouraging degree, and hope of solution was almost abandoned, when thought was given to the rolling temperature of the ingots. Up till this time, although observed, no great stress had been laid on the point, but as the last resort the information was correlated with the other data.

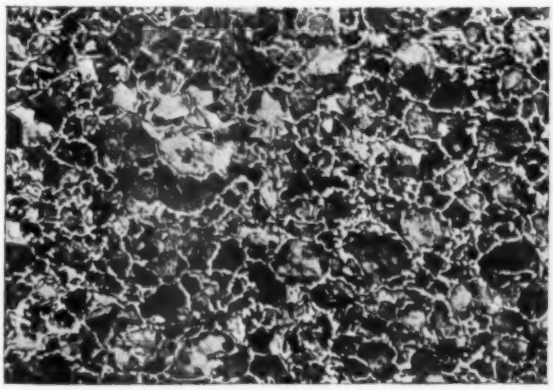


Fig. 1.—Photomicrograph of section rolled from overheated ingot, × 150.

After some months of patient observation covering many casts of steel, the fact gradually became manifest that those ingots which had been rolled from the higher temperatures of 1,300°–1,350° C. were definitely less responsive to heat-treatment than others rolled from below the minimum of that range.

It was therefore decided to promote such conditions in certain ingots, and from the same cast of steel take others for comparison which had not been overheated.

Accordingly, from an ostensibly perfect cast, bearing analysis as given in Table II, a single ingot was taken and soaked at 1,300°–1,320° C. for the same time as that employed for the remaining ingots at the more normal and lower temperature of 1,220°–1,250° C.

TABLE II.
COMPOSITION OF THE NORMAL STEEL USED FOR TRIAL.

	%
Carbon	0·48
Manganese	0·78
Sulphur	0·040
Phosphorus	0·043
Silicon	0·141

A micrographical examination of the rolled section was somewhat disappointing, inasmuch that the expected indications of overheating were not apparent, yet it had been established that steels of this analysis rolled from such temperatures had definitely been of the unresponsive character. Fig. 1 is a photomicrograph of a typical field. It was decided, therefore, to proceed with the trial and manufacture tools to be heat-treated.

A representative number of tools were forged both from the overheated and normal steel and subsequently treated by quenching in fused salts from 800° C. and tempering at 280° C. before submitting to testing.

Three methods of testing may be applied in such cases. Brinell readings and file tests comprise two, which are non-destructive, and a hammer test makes the third, which may be destructive. To all these tests were the trial tools submitted.

File tests were useless, revealing no apparent difference between the two types. Hammer tests were more informative, however; an experienced hammer-tester acquires some considerable skill in the "feel" of a plate which "gives" or fails to give, respectively, according to its nature. Such "give" is really a question of resilience distinct and apart from the impact value, as the latter can yield a relatively high figure in the absence of actual brittleness. Probably the term "give" may be described best as the distance a plate may bend under impact without taking either permanent set (if too soft) or fracturing (if too hard). Application of hammer tests indicated quite clearly that the tools made from the overheated steel were possessed of the "dead" feeling described earlier, although not to the full extent as had been observed on some previous occasions, while the normal tools were perfect in every way.

The "dead" feeling meant that the tools took a permanent set too soon and too easily on hammering, and also were lacking in the resonant ring one learns to expect from a heat-treated plate. In any case, all these three points stood out clearly in comparison with the contemporary series.

To the academic mind a hammer test may be regarded as a method too rough for reliable information, and accordingly Brinell readings were taken by means of a Firth Hardometer loading 30 kg. on a 136° pyramid diamond. Six readings were taken on each tool in corresponding positions, but these were reduced to an average in Table III for ease of assimilation.

TABLE III.

AVERAGE HARDNESS FIGURES ON TOOLS IN TRIAL.

Tool No	136° D.P.N. Loading 30 kg. Normal Steel, Average,		136° D.P.N. Loading 30 kg. Overheated Steel, Average.
1	639	..	598
2	661	..	579
3	589	..	527
4	628	..	579
5	661	..	598
6	639	..	—
Average D.P.N. ..	636	..	576

From the figures in Table III it will be clearly seen that a difference in hardness did exist between the two steels, and such difference was noticeable on hammer testing. An average difference of 60 diamond hardness may not superficially appear much to worry about, but when the individual figures are definitely running lower in the one case than in the other, coupled with the fact that they result from a deliberate attempt at their production, the inference appears established that overheating of the ingots is the underlying cause. Consequently, it was arranged to control the temperature in this direction and the subsequent results have proved unquestionably that the "dead" steel could thereby be obviated.

The story would not be complete without a note on how the material in stock affected in this manner was dealt with. Analysis being right, and no signs of oxidation at grain boundaries, it was felt that the steel *must* sooner or later respond to some treatment or other.

The most obvious method was to normalise before hardening. While an improvement, affected steels were not put entirely right. Double normalising was better still, but not yet correct. Eventually, after much experimentation, it was found possible to deal with the work in three ways. Firstly, by normalising three successive times before hardening. Secondly, by a single close anneal at 680° C. and furnace cooling, followed by single normalising at 780° C., and thirdly, by quenching in water from two separate heatings to 760° C., followed by the normal salt-bath quenching from 800° C. Each method, however, was costly and to be deprecated, but represented the only alternative to scrapping good forgings, and actually the application of the last described resulted in a 95% successful result. With prevention as ever being better than cure, however, co-operation with your steel supplier in cases of this nature is always the strongest suit.

Valuable Deposits of Brucite

THE award of the Professional Institute Medal to Mr. M. F. Goudge, B.A., B.Sc., of Ottawa, Mineral Technologist of the Bureau of Mines, Department of Mines and Resources, is a recognition of his discovery of valuable deposits of brucite, and for developing a process for its concentration and making it possible to obtain pure calcined brucite or magnesia at a low-production cost. This contribution to metallurgical science has also made possible the development of limestone deposits which previously had relatively small economic value, thus substantially augmenting Canada's known mineral resources.

A practically unlimited supply of magnesia of the highest grade has been made available as a result of Mr. Goudge's discovery of brucite limestone in the Gatineau and Bryson areas of Quebec, and Rutherglen, in Northern Ontario. In addition to its use in refractories, magnesia is important in various industries, including the manufacture of thermal insulating materials, in a process of sweetening petroleum distillates, in the compounding of rubber, etc.; but of greatest importance is its use in the manufacture of magnesium metal, particularly by the electrolytic process.

Almost equal in importance is the method invented by Mr. Goudge for the recovery of high-grade granular magnesia, together with marketable by-products, from this brucitic limestone, and the perfection of the technique of the process in a pilot plant at the Bureau of Mines at Ottawa. Further, Mr. Goudge has been untiring in his assistance to industry in translating the pilot plant practice into commercial proportions to supply the much needed magnesia required for industry, especially under present conditions, when the need is greatest and supplies from outside sources greatly limited. Already a large new industry has been established to develop the brucite resources, and more industries using magnesium metal are expected to follow.

Exhaustive tests made in Canada, Great Britain and the United States on the granular magnesia produced from this brucitic limestone have proved it to have unique and extremely valuable chemical and physical properties. Magnesia bricks made from brucite at the pilot plant have been tested as a lining for large reverberatory furnaces in metallurgical plants, and have been found to withstand normal service conditions twice as long as those made from the best imported material available.

Industrial experimental trials in England and Canada have indicated that the use of granular brucite magnesia will make possible a saving of from 15 to 20% in the cost of producing magnesium metal. As a result of the success achieved, the British and United States Governments are investigating the possibilities of deposits of brucitic limestone in the Island of Skye and in Southern United States.

Normally, the Professional Institute award one medal annually for a contribution of outstanding merit by the Civil Service. This year, however, two medals have been awarded so that the difficulty of deciding between an administrative and a scientific contribution has been avoided. The medal for the outstanding administrative contribution has been awarded to Mr. John Patterson, B.A.Sc., M.A., of Toronto, Controller of Meteorological Service, Department of Transport, for his long and brilliant career in scientific work and administration, which has earned for him universal recognition, and for his contribution to Canada's war effort by meeting the operational needs in weather forecasting of the R.C.A.F., the British Commonwealth Air Training Plan, the Trans-Atlantic Ferry Command, and also the Royal and Royal Canadian Navies.

The Damping Capacity of Engineering Materials

Under many conditions of service metal components are subjected to the effects of vibrations, especially in various rotating and reciprocating masses, such as turbines, reciprocating engines and the like, with their own operating speeds. The capacity of a metal to cause these vibrations to cease is known as its damping capacity. This phenomenon is admirably discussed in a recent paper, an abstract of which is given here, in which a new testing apparatus is described and results tabulated of a large number of tests on damping capacity of materials at room and elevated temperatures.

IN recent years the damping capacity of metals has been the subject of considerable study, mainly because of its direct practical importance. It is fairly well known that metals, under many conditions of service, are subjected to the effects of vibrations set up by resonant conditions. The capacity of the metal to cause these vibrations to cease is known as its damping capacity, and this capacity has an important influence on the service life of metal components, for this reason increasing attention is being given to the subject. The terms " damping capacity " and " mechanical hysteresis " have come into use in recent years to cover the energy losses which occur within the materials of a structure when they are subjected to fluctuating stresses, and it is with this phenomenon that a recent paper by Dr. W. H. Hatfield, G. Stanfield, and L. Rotherham* is mainly concerned. These internal losses in the material may be regarded as being due to imperfections in the elastic properties of the material.

If a material is stressed beyond the yield-point—i.e., up to a stress where plastic yielding occurs, energy is dissipated as heat. In general, engineering design has aimed at keeping the stresses below the point where such effects take place. In a truly elastic body, one has been led to expect that not only is strain proportional to stress, but also that on removal of the stress the material retraces the stress-strain relationship existing during the application of the stress. If this expectation were realised during a cycle of alternating stress, the strain energy would be fully recovered at each removal of stress. It has now been established that even over a range of stress which by usual methods of determination is considered to be within the elastic limit, some energy is lost. A precise determination of the stress-strain curves for stress cycles over quite low values of stress gives a " hysteresis " loop, the area of which denotes the energy lost per cycle. The " damping capacity " of the material is taken as the ratio of this energy loss per cycle to the full strain energy at the maximum stress of the cycle. A loss of energy in this way occurs at stresses well below the fatigue limit of the material, and can apparently continue for an indefinite number of repetitions of the stress.

It is recognised that in most mechanisms where vibrations occur the damping introduced due to the properties of the materials employed is, in general, only one factor contributing to damping. Nevertheless, a knowledge of the characteristics of structural materials in this respect is obviously of some importance, in order that an assessment can be made of its influence on the behaviour of the mechanism as a whole and further to decide to what extent the choice of the material to be employed for a particular purpose should be guided by its damping properties.

The cases calling for consideration are those where, owing to the speed of rotation or oscillation or other special feature of an engineering component, repetition of impressed forces occurs at particular frequencies which coincide or nearly coincide with the natural frequencies of vibration of the component parts. Under these circumstances, resonance may occur—i.e., the amplitude of the natural vibration is built up and the movement may quickly reach serious and dangerous amounts. Damping, from whatever cause, naturally acts in restricting the extent of such vibrations.

One type of example which has received much attention in recent years is the crankshaft of reciprocating engines. A long and flexible shaft, such as occurs in multi-cylinder engines, can undergo a complex system of vibrations—suffering torsional, direct torsional and compressive as well as bending stresses. The torque developed, and the various operating pressures are periodic, and any synchronism, or near synchronism, between the frequency of the impressed forces and one of the natural frequencies of vibration of the shaft and its attached masses can give rise to vibration and strain substantially in excess of those which would exist if the forces were only static. The statement has been made that it is safer to use, for certain types of crankshaft, a steel of relatively low fatigue limit, which has a high damping factor rather than a high tensile steel of low damping factor, even though the latter has a higher fatigue limit. On the other hand, Shannon† has deduced that the degree of damping contributed by the crankshaft material in cases which he examined is relatively insignificant, and that the chief damping comes from the bearings and the films of lubricant.

For ships' propelling shafting, the chief contribution to damping is apparently the effect of the fluid acting on the propeller. Other cases of importance in engineering are propeller and fan blades, turbine parts (particularly turbine discs and blading), high-speed shafts carrying rotating masses with various degrees of " balance," such as turbine shafts, certain machine tools and centrifuging machinery. A different class of problem arises where flexible parts, such as aeroplane wings and overhead cables, etc., are subject to wind effects, although additional disturbing factors, such as are received from adjacent rotating parts, may be present. Frame structures, such as buildings, bridges, etc., whether welded or riveted, may be subjected to vibration from various influences—a much quoted example being that of a regiment of soldiers marching over a bridge in step. Föppi discusses the conditions arising in a number of engineering applications.

The features contributing to damping vary considerably in the different cases: thus it will be recognised that a welded frame may lack the frictional damping of a similar structure made up by riveting. Detailed mathematical analysis has been made of many systems and mechanisms starting from a freely vibrating system as modified by a regularly impressed force and further modified by certain types of damping. As long as the various factors are properly known, a mathematical solution is usually obtainable. One interesting feature is that unless the damping effect is substantial the frequency of vibration of a system is not measurably affected by it. An interesting treatise on the general subject is that by J. P. Den Hartog†, of Harvard University. An instructive diagram is to be found in that

* North-East Coast Inst. Eng. and Shipbldrs. Advance Copy.

† " Mechanical Vibrations." McGraw Hill, 1940.

work, for a simple vibrating system. The frequency of the impressed force—representing the running speed of the system, and expressed as a ratio to the natural frequency of the system is plotted horizontally, while the amplitude of vibration is plotted vertically. The several curves correspond to different degrees of viscous damping—each expressed as a ratio to the " critical " damping—i.e., the amount of damping which would just prevent any oscillation at all after an initial displacement. The curves show that when the applied frequency is near to the natural frequency the amplitude reaches very high values unless substantial damping forces are present.

The subjects of vibration and resonance effects, certainly present problems to the engineering designer. If in a particular design it is known that serious vibration occurs there are several courses open to him. One method is to modify the natural frequencies of the various parts concerned in relation to the working speed by changing the dimensions—the stiffness—and the masses of the parts. The case of a shaft which normally runs at a speed above a " critical speed," is not so easily dealt with since in working up to speed from rest, the critical speed has to be passed through if only momentarily. The engineer may introduce special damping mechanisms (as in the case of the motor-car shock absorber). He may alter his working speed. He may sub-divide his structure into separate units. He may in special cases seek to alter his structural materials, and it has been claimed that increase in the damping capacity of the material is obtainable at some sacrifice of mechanical strength, including resistance to fatigue, with the attendant necessary increase in sizes of components. Careful consideration of the various factors involved is clearly necessary for each individual case.

Recorded experimental work on damping capacity dates back to Thomson (Lord Kelvin), who observed in 1865 that the " viscosity of metals " had a higher value than might be expected on simple thermodynamic reasoning. Since that time the number of papers published on the subject is large, but so far the results have not been given a simple physical interpretation as has been done by Andrade in his theory of liquid viscosities. This gives both the order of magnitude of the viscosity of liquids, and its variations with temperature. In solids the variation with temperature is more complex and the variations in damping caused by relatively slight alloy additions comparatively large.

The earliest measurements of damping capacity in the present work were carried out on a Cambridge Instrument Company's torsional damping recorder. With careful use it is possible to obtain reasonably reliable results which, although they are generally a little higher than those obtained with a later machine, designed by the authors, show the same kinds of variation in different steels. In designing this new machine, which is described, the authors decided to use optical recording to eliminate the possibility of error caused by friction in other recording machines.

The accuracy of the results of tests taken with this machine is considered to be of the order of about 2·5%, and the difficulty is chiefly one of measuring the record obtained. This record was projected in a special machine at a magnification of 10 to 1, and with all precautions it was not possible to increase the accuracy much beyond the order stated. This, however, was sufficient to indicate that the reproduceability of results was good, and a higher accuracy was hardly necessary, since it is known that very slight influences can affect the damping considerably. The authors are of the opinion that the results obtained from a considerable number of tests place the damping of the materials tested in their relative orders, and give an indication of the effects which might be expected in service.

Tables of test results given in the paper have been sub-divided for convenience. These results include tests on mild steels, alloy steels, rust-, acid-, and heat-resisting steels, cast iron, sintered carbides, while other metallic materials tested included nickel, cobalt, nickel-copper and nickel-iron alloys ; several magnesium base and aluminium-base alloys ; a range of copper-base alloys ; lead ; and solder ; while records are also given for glass, ivory, ivorine, and several types of bakelite. The damping capacity of the majority of the materials was tested at room temperature and at elevated temperatures. Examples have been observed of both increases and decreases in damping with increase of temperature, while in other cases over a wide range of temperature changes in damping are comparatively slight. In the same way, variation with stress can be either an increase, a decrease or only a very slight change. The truly austenitic alloys show relatively low values of damping, while the stainless materials of the 13% chromium type give particularly high values. Low-carbon steel shows an intermediate value.

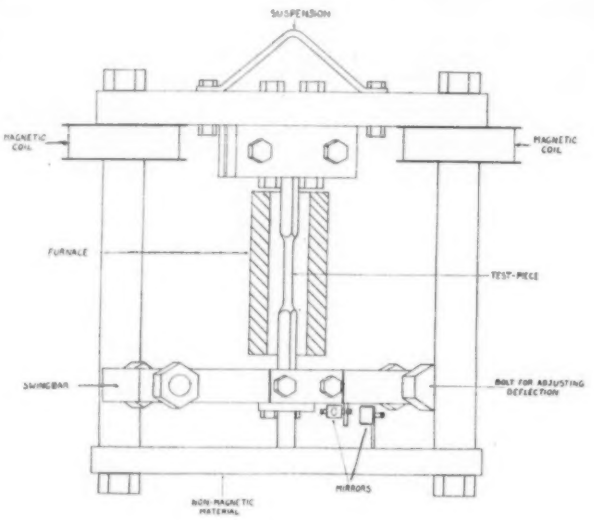


Fig. 1.—Elevation of new apparatus for testing damping capacity of materials.

Comparison of the results presented in the different tables is interesting. For instance, the values on sintered carbides while a little higher than for cobalt, one of the constituents in the alloys, are nevertheless of the same order of magnitude. It seems very likely that a hard chemical compound, such as tungsten carbide, will not have a very high damping capacity, and the cobalt may be responsible for most of the damping recorded. Following the same reasoning, the cementite occurring in carbon and alloy steels may not contribute a great deal to the damping, although obviously in this case the relative amounts of carbide and matrix is very different. However, the 0·26 and 0·36% carbon steels show lower damping capacities than the 0·09 and 0·10% carbon steels. The distribution of constituents and their state of strain most probably have important influences.

The exceptionally high values of damping capacity observed in nickel at room temperature are remarkable. The crystal structure of nickel is similar to that of the 25% chromium, 20% nickel alloy, yet this alloy, unlike nickel, has a particularly low damping capacity. Nickel has distinctive ferro-magnetic properties, unlike the 25% chromium—20% nickel alloy which is non-magnetic, and it is noted that other austenitic alloys which are slightly magnetic have higher damping capacities than the 25% chromium 20% nickel alloy.

From the results on cobalt, nickel and the 36% nickel-iron alloy also, it would appear that magnetic influences are important, but beyond this the authors are not able to suggest an explanation of the results obtained.

The Influence of Tin on Alloy Steels*

By G. R. Bolsover and S. Barraclough

The general effect of tin on the properties of steels in the hardened and tempered condition and of molybdenum in counteracting that of a high tin content is shown, and further data indicate the influence of tin on a nitriding steel after a blank nitriding operation and on a case-hardening steel in the quenched condition. In this abridgement the results are shown in the form of graphs.

A CAST of alloy steel with impact values lower than normal led to an investigation into the cause, from which it was concluded that it might be due to a content of tin greater than would usually be present. As no data on the effect of tin on alloy steels was available, it was decided to make a brief survey of the influence of tin on some of the alloy steels most commonly used. The steels selected were the manganese-molybdenum, 3·5% nickel, and 3% nickel-chromium qualities as representative of steels for oil-hardening and tempering, with a 0·35% carbon steel, without any alloying element, included as a basis for comparison. A 4·5% nickel-chromium case-hardening steel was also included.

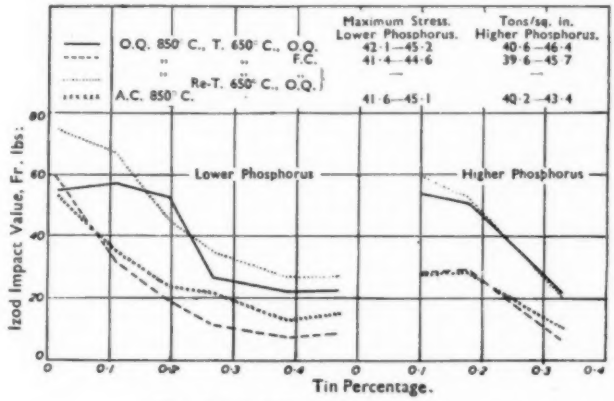

Maximum Stress. Tons/sq. in.
Lower Phosphorus. Higher Phosphorus.
O.Q. 850° C., T. 650° C., O.Q. 42·1—45·2 40·6—46·4
" " F.C. 41·4—44·6 39·6—45·7
Re-T. 650° C., O.Q.
A.C. 850° C. 41·6—45·1 40·2—43·4
Lower Phosphorus
Higher Phosphorus
Izod Impact Value, Ft. lbs.
Tin Percentage.

Fig. 1.—0.35% carbon steel.

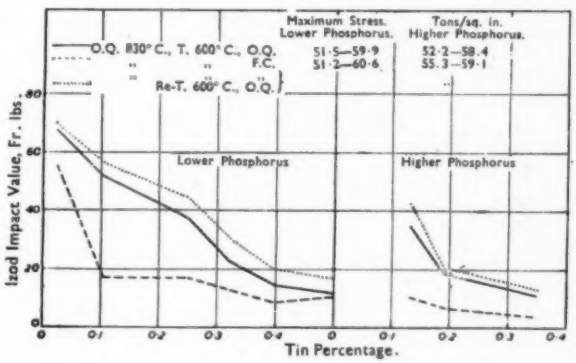

Maximum Stress. Tons/sq. in.
Lower Phosphorus. Higher Phosphorus.
O.Q. 830° C., T. 600° C., O.Q. 51·5—59·9 52·2—58·4
" " F.C. 51·2—60·6 55·3—59·1
Re-T. 600° C., O.Q.
Lower Phosphorus
Higher Phosphorus
Izod Impact Value, Ft. lbs.
Tin Percentage.

Fig. 3.—3.5% nickel oil-hardening steel.

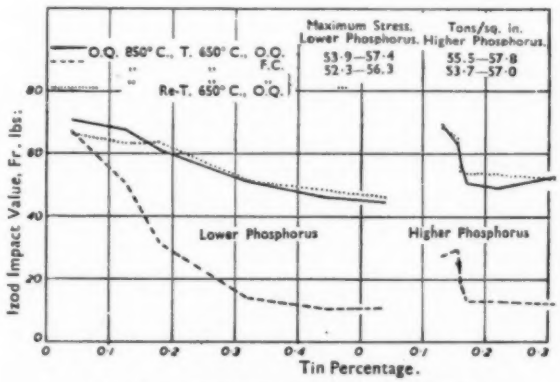

Maximum Stress. Tons/sq. in.
Lower Phosphorus. Higher Phosphorus.
O.Q. 850° C., T. 650° C., O.Q. 53·9—57·4 55·5—57·8
" " F.C. 52·3—56·3 53·7—57·0
Re-T. 650° C., O.Q.
Lower Phosphorus
Higher Phosphorus
Izod Impact Value, Ft. lbs.
Tin Percentage.

Fig. 2.—Manganese-molybdenum steel.

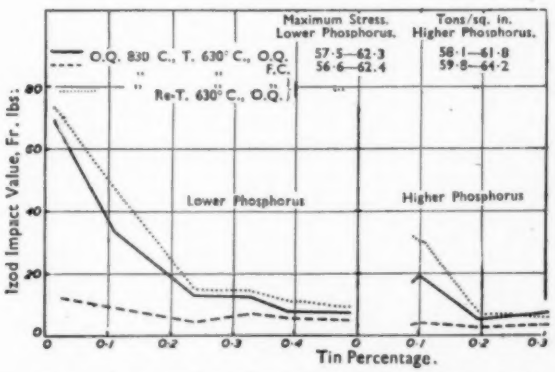

Maximum Stress. Tons/sq. in.
Lower Phosphorus. Higher Phosphorus.
O.Q. 830° C., T. 630° C., O.Q. 57·5—62·3 58·1—61·8
" " F.C. 56·6—62·4 59·8—64·2
Re-T. 630° C., O.Q.
Lower Phosphorus
Higher Phosphorus
Izod Impact Value, Ft. lbs.
Tin Percentage.

Fig. 4.—3% nickel-chromium oil-hardening steel.

Series of the types of steels were accordingly prepared in a 35 kva. high-frequency furnace of 18 lb. capacity, of the Ajax Northrup spark-gap type, producing ingots of approximately 2 in. square section, these being forged to $\frac{7}{8}$ in. dia. bar for heat-treatment and testing. Each series of steels included a basis steel with a low tin content, the other samples having an addition of 0·1, 0·2, 0·3, 0·4 and 0·5% of tin. Further, the effect of phosphorus was investigated by the inclusion of a limited number of steels aiming at a phosphorus content of approximately 0·045%, as compared with a percentage of the order of 0·020 in the main series.

The early results were of sufficient interest to promote queries regarding the influence of molybdenum, if present, for example, in a steel of the 3% nickel-chromium type, in conjunction with a high tin content. Two series with approximately 0·2% and 0·4% of molybdenum respectively were therefore produced over a limited range of tin additionsl In order to include a steel of the nitriding type, severa. steels of the 3% chromium-molybdenum quality were also made and examined in the same way.

It will be appreciated that the results recorded can be regarded only as a very general survey of the effect of the presence of tin over the alloy-steel field. It is felt, however, especially in view of the absence of other published data, that the publication of these results will be of some interest and value, not only by preventing the repetition of similar work, but by indicating the direction in which further investigation may be made.

The results obtained for the various steels are recorded in eight tables, and are also shown in the form of graphs, which are reproduced in Figs. 1 to 8. As will be appreciated, in the manufacture of nearly seventy different samples, slight variations from the composition required are liable to occur. All the steels, however, were tested, and all the results,

* Iron and Steel Institute Paper. Advance copy, May, 1942.

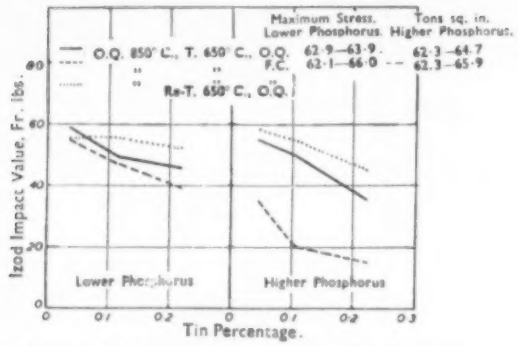

Maximum Stress. Tons sq. in.
Lower Phosphorus. Higher Phosphorus.
O.Q. 850° C., T. 650° C., O.Q. 62·9—63·9 62·3—64·7
F.C. 62·1—66·0 62·3—65·9
Re-T. 650° C., O.Q.
Izod Impact Value, Ft. lbs.
Lower Phosphorus
Higher Phosphorus
Tin Percentage.

Fig. 5.—3% nickel-chromium-molybdenum oil-hardening steel (0.20—0.25% molybdenum).

even though certain casts had one or more elements outside the desired range of composition, are reported, but for the graphs only those steels within fairly narrow limits have been selected. To decide whether a steel should be included in a graph, judgment was based more on the tensile strength than on detailed composition, provided that the latter was within reasonable limits of that desired. In view of this, and in order to indicate readily the degree of variation, the range of maximum stress for each type of steel in each condition of heat-treatment has been stated on the graphs.

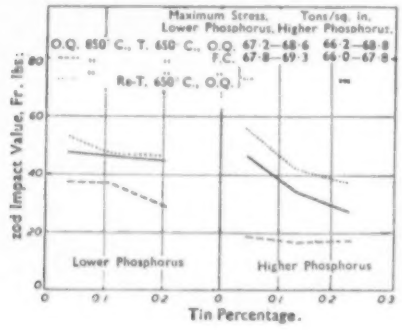


Fig. 6. 3% nickel-chromium-molybdenum oil-hardening steel (0.4—0.5% molybdenum).

The general scheme of heat-treatment has been as follows :—

For steels to be hardened and tempered—

1. Oil-quench and temper, followed by oil-quenching.
2. Oil-quench and temper, followed by furnace-cooling. (Furnace cooling occupies approximately 9 hours from the tempering temperature to the temperature of 300° C.)
3. Oil-quench and temper, followed by furnace-cooling, followed by a re-temper at the same tempering temperature and oil-quenching.

Additions to this programme were made in the case of the plain carbon steel by including tests in the normalised condition, and in the case of the 3% nickel-molybdenum steel by including tests after a dummy nitriding treatment. The case-hardening steel was tested as oil-quenched from 760° C. and tempered at 150° C.

Commentary

On all the hardened and tempered steels examined, the influence of the presence of tin at even 0·1% is shown on notched-bar impact tests. It is interesting to note in the case of the basis carbon steel that this effect is not so marked in the condition oil-quenched after tempering as in the normalised condition. On the alloy steels the effect of tin additions appears to be counteracted very appreciably by the addition of molybdenum, as shown by the results on the manganese-molybdenum steel and on the 3% nickel-chromium-molybdenum steels, comparing these with either the 3% nickel or the 3% nickel-chromium (without molybdenum) steels.

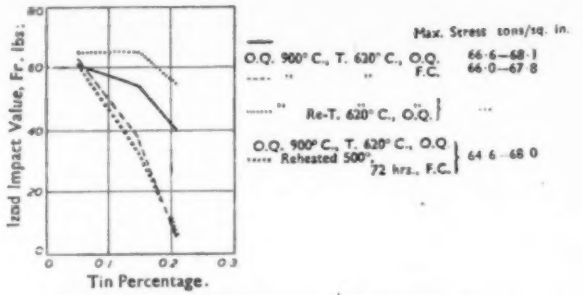

Max. Stress tons/sq. in.
O.Q. 900° C., T. 620° C., O.Q. 66·6—68·1
F.C. 66·0—67·8
Re-T. 620° C., O.Q.
O.Q. 900° C., T. 620° C., O.Q. Reheated 500°, 72 hrs., F.C. 64·6—68·0
Izod Impact Value, Ft. lbs.
Tin Percentage.

Fig. 7.—3% chromium-molybdenum steel.

The higher phosphorus steels in general, give lower results than the lower phosphorus steels, but on the data available this appears to be more in the nature of a lowering of the impact value owing to the phosphorus content rather than due to any increasing effect of tin on the high-phosphorus as compared with the low-phosphorus steels.

The effect of tin on the nitriding steel again appears to be counterbalanced to some extent by the addition of molybdenum, but the lowering of the impact value after nitriding in the presence of tin is very marked. The 4% nickel-chromium case-hardening steel suffers only a slight decrease of impact value up to 0·15% tin, but with higher contents the values fall seriously.

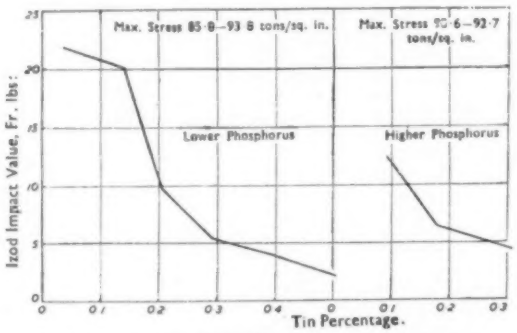


Fig. 8.—4.25% nickel-chromium-molybdenum case-hardening steel.

Non-Ferrous Scrap Metal in Canada

ALL dealings in scrap zinc, copper, tin, brass, aluminium and other non-ferrous metals are now under strict control in Canada. An order has been issued which is designed to ensure that all non-ferrous scrap is channelled into war production, or into essential production related to war. All non-ferrous scrap dealers buying or selling scrap to smelters, foundries or ingot makers must obtain a licence. Smelters and ingot makers must also be licensed. Foundries will require no licence, but must buy only from licence holders.

Control of Electrical Heating Circuits

THE conventional method of controlling the input of electric current to heating processes is by the use of a resistance or potentiometer. An alternative method is to regulate the average amount of electrical energy supplied in a given time by periodically interrupting the current. By arranging a suitable on/off time ratio the amount of energy consumed can be varied at will from zero to full load. Apparatus for this purpose, known as the "Energy Regulator," with an average cycle time of 30 secs. is suitable for adjusting power input to heating loads and keeping it constant. Full technical details are given in a leaflet obtainable from Sunvic Controls, Ltd., 1, Kingsway, London, W.C. 2.

The Grinding of Carbon and High-Speed Steels

By A. J. Schroeder, D.Sc. (Eng.)

In the machine shop, speed in production is largely dependent upon the condition of the cutting tools used; they must be maintained in an excellent condition to obtain maximum result, and this necessitates careful grinding to meet the requirements of the materials to be machined. In this article attention is directed to the grinding of tools for turning, planing, and automatic lathe operation.

IN order to obtain satisfactory work on centre lathes, capstan or turret lathes, planers and automatics, it is necessary to grind the cutting steels correctly. The tools mentioned are made from different materials according to their use, either from carbon steel or high-speed steel or cemented hard carbide. In this treatise only the grinding of the carbon and high-speed steels will be dealt with.

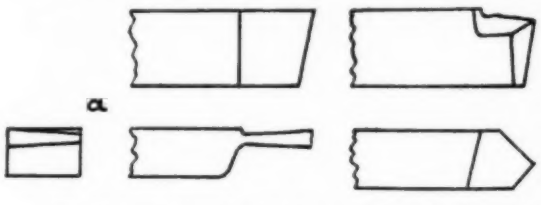


Fig. 1.—Shapes of tools for turning, planing and automatic lathe operation.

The shapes of tool for turning, planing, and automatic lathe operation are very different. They differ in regard to the shape of the cutting edge: possessing either a parallel or slanting edge (Fig. 1). In the one category the cutting edge is parallel to the base of the shank (*a*), whereas in the other the edge forms an angle on the tools with a slanting edge (*b*). Another point to be observed is in regard to the nose of the tool, which may be either straight or curved. Tools with a straight nose can be ground easily, whilst the grinding of the tools with a curved nose, as in the case of forming tools, raises certain difficulties. The description of the angles and edges on the tools is shown in Fig. 2.

In cutting-tool grinding a distinction must be made between the grinding of a new tool and re-grinding after use. When not bought out, new tools are ground in the tool-room according to drawings. Care must be taken that the tools are well prepared and that the cutting planes have the correct angles. This is especially important for high-speed steel-tipped tools, so that the tips sustain the minimum loss in material, and they require less frequent re-grinding.

High-speed-steel turning and planing tools are often ground in two stages. In this case the first operation can be "dry." This has the advantage of being time-saving; moreover, cracks do not form in the steel surface. Such cracks (which are barely perceptible to the naked eye) form if the tool, heated by the grinding friction, is suddenly cooled by immersion in cooling water. This danger decreases in hardened tools if ample coolant is provided and the tool is lightly pressed against the grinding wheel. Furthermore, the allowance of material for grinding after hardening must be as small as possible. It is, however, not sufficient merely to burnish the hardened tool, as this would leave the burnt layer caused by hardening to be only glossed over, therefore enough material, up to 0·04 in., must be left to allow for grinding. With high-speed steels, which have a tendency towards heavy scaling, and even, occasionally, to slight burning away, it is necessary sometimes to grind as much as 0·04 in. away to obtain maximum efficiency.

With some cutter grinding machines the grinding operation is done on the periphery of the grinding wheel. This has the disadvantage that the cutting planes assume the curvature of the grinding wheel—i.e., become hollow ground, instead of with a flat surface. In such cases the edge produced is not capable of offering as much resistance as a flat ground edge. Such tool grinders are, therefore,

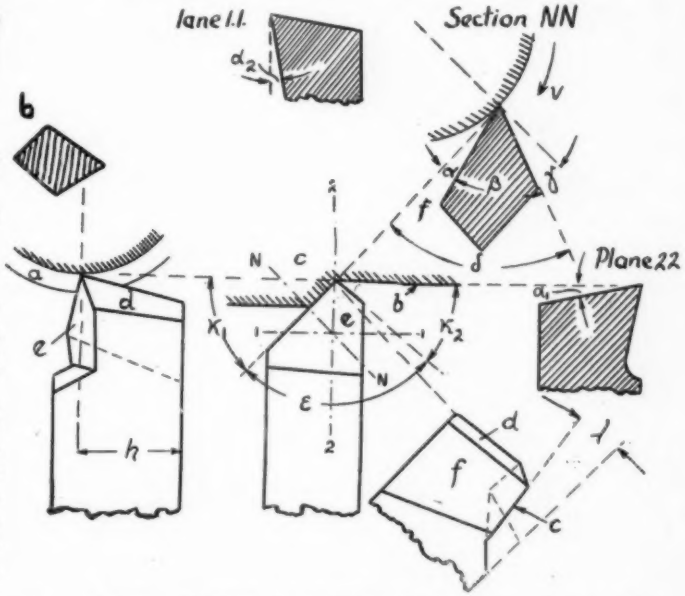


Fig. 2.—Showing the angles and edges on various tools.

a = Clearance angle. (Perpendicular to cutting edge.)
a_1 = Front clearance angle.
a_2 = Side clearance angle.
β = Tool angle.
γ = Top rake angle.
δ = Real cutting angle = $a_1 + \beta$.
ϵ = Angle at point.
κ_1 = Cutting angle in plan. (Setting angle.)
κ_2 = Back setting angle.
λ = Back rake angle.

a = Cutting plane.
b = Machined surface.
c = Main cutting edge.
d = Clearance plane of back cutting edge.
e = Top rake plane.
f = Clearance plane of main cutting edge.
h = Height of cutting point.
v = Cutting speed.

provided with large grinding wheels, usually 20 to 24 in. in diameter. In front of the grinding wheel there is a grinding pad, which is adjustable to the decreasing diameter of the grinding wheel.

The cooling appliances on these machines are in various positions. On some products a water-trough, adjustable in height, is built into the machine column. This water-trough is lifted by pressure on a foot pedal, so that the grinding wheel is immersed and carries the cooling water to the point of grinding contact when the grinding wheel is in motion. Other producers of such machines provide them with a special cooling pump, which leads the water either to the grinding place through the pierced grinding pad or to the grinding wheel inside the guard.

Tool grinding on this type of machine necessitates skill, as it is left to the operator to bring the tool into the correct position for obtaining the cutting angles required. Before the correct shape of edge is reached, it must be repeatedly inspected. All this is laborious, and takes much time.

Better results are obtained with cup wheels. The tools are ground on the face of the grinding wheel, and thereby flat cutting planes are produced. A further advantage is that these machines have two grinding pads with dogs, which can easily be clamped and adjusted by means of a scale to any angle desired. The direction of rotation of the grinding spindle is reversible. Wear on the grinding wheel is equalised by adjusting the grinding spindle so that the grinding wheel can be entirely used up. Such a machine can grind dry or wet for rough and finish grinding.

In shops where large numbers of cutting tools, especially those with larger shank sections are used, it is best to work with automatic tool-grinding machines. The tools are clamped into the tool post, which can be adjusted to the grinding wheel in two directions, and to whatever angle is desired. Grinding contact is made when either the slide of the grinding wheel is moved to and fro and the tool post is fixed, or vice versa, the grinding spindle is kept in one position, while the tool-box is swung about it. A cup wheel is used and the feed is effected automatically or by hand. On these tool grinders scales are fitted from which the adjustment of angles for the post tool-holder can be read off.

The cutting planes on turning and planing tools can also be ground out of the solid by machines of this type. This has the advantage that the shape of the edge need not be forged and milled previously. Only curved cutting tools necessitate bending. The cutting form is also ground out of the solid. The tools must be hardened before this roughing, and should be ground dry. The finish grinding takes place after hardening in the same way, using an ample coolant. Wear on the grinding wheel is heavy if the tool be ground out of the solid, unless a suitable grinding wheel is used. Therefore, care must be taken that the saving of preliminary work on the tools is not offset by excessive grinding costs.

The top-rake plane is ground hollow on roughing tools for heavy duty, because consequently a better chip flow is attained, and therefore heavier feeds can be used (Fig.3). Hollow top-rake planes can easily be obtained on automatical tool grinders by using a suitable attachment with which these machines are provided, or for this purpose alone special machines are available.

Hand-tool grinders or electric hand-grinding machines are used for the manual sharpening of blunt turning or planing tools. The electric machines have a grinding wheel on each end of their spindles for rough and finish grinding. They have adjustable grinding pads, one of which is generally adjustable to different angles. The small diameters of the grinding wheels are, however, unsatisfactory because of their hollow grinding propensities. A further disadvantage is that these electric tool grinders have no adequate provision for water-cooling the work.

The necessary attention is often not given to the sharpening of tools in the workshops, with the result that they are frequently overheated, so that the required efficiency can no longer be obtained. Therefore, the grinding of the blunt tools should be transferred to the tool-room, where they will be changed for sharp ones at the tool stores. This does not mean that no grinding work at all may be done by machine hands, and that all tool grinders can be removed from the machine shops. Grinding of small radii on the tools, sharpening of thread tools, small boring tools, etc., can be left to the operator's experience, and to the demands of the work in question.

The simple cutting tools for automatic machines, such as tangential, side, boring, parting off, etc., are chiefly manufactured as straight forms without any cutting planes ground to definite shapes, so as to allow them to be ground by the buyers to suit themselves in the matter of individual angles of cutting planes, and for the class of metal they will have to cut, as well as the adaptation of form, for the type of component. Grinding of these tools is always done by tool setters on small hand-grinding machines. In order to avoid undue idleness on the part of the automatics, by the time taken to grind the tangential tools (which are roughing tools, and therefore frequently blunted) they are advantageously provided with two edges on each end so that there are four edges on every tool. In this way the tools can be changed four times. The correct grinding of these tools is especially important for machining hard and tough materials, because in that way long turnings with a small spiral are produced which escape easily without choking the machine.

Fig. 3.—Roughing tool for heavy duty.

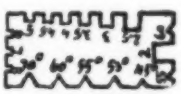


Fig. 7.—Gauge for cutting form fo thread tool.

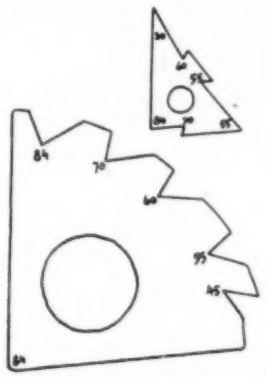


Fig. 4.—Plate gauge for testing tool angles.

Great importance must be attached to the sharpening of the former tools, as their profile changes when they are incorrectly ground. They should, therefore, always be sharpened only in the tool room. Surface grinding machines are best adapted for the grinding of flat formers, in which case the tool is held by a magnetic holding plate. Circular form tools especially should have particular attention paid to their grinding, both by reason of their cutting efficiency and with regard to the trueness of form imparted to the components by these tools. These particular types of form tools are best ground by placing them on a mandrel, which in turn is clamped in an adjustable block, which details are included in the equipment of all universal tool grinders; the cutting plane must be lined up according to the angle and be brought to the face of the cup wheel. The feed motion takes place through gradual rotation of the toe spindle. If many such tools are to be ground, the application of a special device is justified, one provided with a worm gear and a scale for the feed motion. Tool groups as used, for example, for slotting the grooves in pistons, are best ground in an assembled state, to avoid the trouble and time in the individual adjustment of the tools. In this case it is necessary that such groups are provided with a suitable holder in order to grind them.

To obtain smooth work surfaces by using turning and planing tools, and if these tools are to give long service, it is necessary to grind them in a proper manner. The quality of the grinding depends entirely on the selection of the correct grinding wheel. Vitrified bond grinding wheels with a grain size of between 24 and 80 are used for cutting tools made from carbon and high-speed steels. Wheels with grain sizes from 36 to 46, and a designation of grade from N to P are suitable for hand-grinding machines, on which medium-sized tools are to be ground. Definite information on the selection of grinding wheels is best obtained from the grinding wheel manufacturer. The grinding wheel must not be too hard, and it must be clean—that is to say, free from dirt and grease.

Attention must be paid to the fact that the feed pressure should be light for hand-grinding, for if the pressure be too heavy grains break off the wheel without their having removed any metal from the work, apart from the fact of having damaged the wheel, often beyond redemption. Furthermore, the tools are overheated if ground thus, despite their water cooling. Therefore, tools should be moved lightly to and fro on the grinding wheel, and only easy feeding of the tools across the wheel gives a good finish to the grinding work and a correct edge.

Coarse-grained grinding wheels with soft grade are used for roughing operations on automatic tool grinders. Wheels with grain sizes from 24 to 36 and grades of from K to M are used according to the size of the tool, and if they be soft or hardened tools to be roughed. Wheels with grain sizes from 46 to 80 and grades J to M are suitable for finishing. The feed-motion must not be too great, for if it is, the grinding wheel will wear away much more quickly (without removing more metal) than if the correct, fine feed is used. The above remarks concerning inadmissible overheating of tools apply here also.

Plate gauges should be used for inspecting the cutting angles on the turning and planing tools (Figs. 4 and 5). The angle sections of the gauges serve to compare the tool angles, while the plain lateral planes of the gauges are used for checking the side clearance angles (Fig. 6). The cutting forms of thread tools are inspected by gauges as shown in Fig. 7. A gauge with a plug and a corresponding measuring surface is useful for measuring the cutting planes on circular form tools. All the gauges mentioned should be part of the equipment of every lathe-hand for the correct measurement of tools in order to eliminate the "eye" measurement of important cutting angles, as is, unfortunately, often the case in workshop practice. Only then, when tools are ground in the right manner, can good results be obtained.

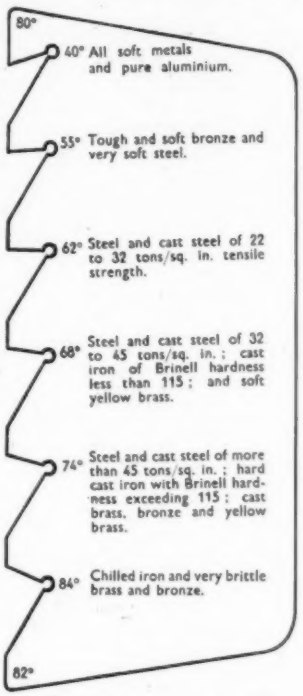

80°
40° All soft metals and pure aluminium.
55° Tough and soft bronze and very soft steel.
62° Steel and cast steel of 22 to 32 tons/sq. in. tensile strength.
68° Steel and cast steel of 32 to 45 tons/sq. in.; cast iron of Brinell hardness less than 115; and soft yellow brass.
74° Steel and cast steel of more than 45 tons/sq. in.; hard cast iron with Brinell hardness exceeding 115; cast brass, bronze and yellow brass.
84° Chilled iron and very brittle brass and bronze.
82°

Fig. 5.—Plate gauges for testing tool angles.

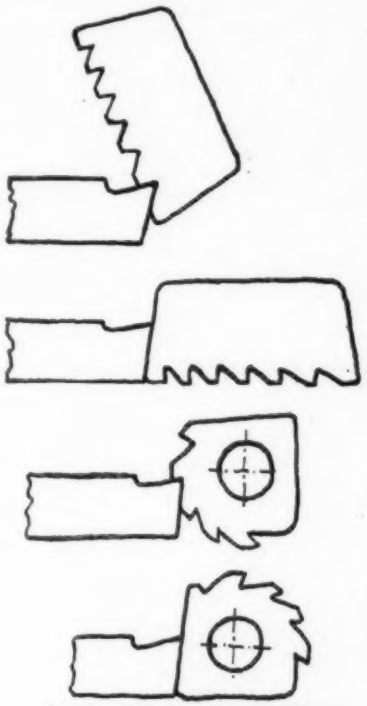


Fig. 6.—Gauges for checking clearance angles.

Durability of Gears

Two reports have been prepared by H. D. Mansion and published by the Institution of Automobile Engineers, dealing with the durability of gears. Thefirst reports an investigation on lubricants, while thesecond is concerned with gear steels.

In the investigation of gear lubricants gear-testing machines, developed by the Institution, were used for testing a number of oils and extreme pressure addition agents, the latter being all added to one standard base oil. The addition agents, or dopes, included both the mild type, suitable for use in engines, and the more powerful type, suitable for hypoid axle lubrication.

After several attempts, a testing technique was developed in which failure of the lubricant film took place, resulting in scuffing of the gears, but it was found that the profile shape and the quantity of oil supplied had a great influence on the susceptibility to scuffing, loads beyond the scope of the machine being carried successfully even with undoped oils when adequate profile relief and a copious supply of lubricant was provided. The main tests described were, in consequence, made on gears with almost unrelieved involute profiles, and with a restricted supply of lubricant.

All of the dopes tested improved, to some extent, the load-carrying capacity of the base oil, though some of the mild dopes were rather uncertain in the improvement afforded. The best dopes of this type tested—viz., a 0·5% concentration of naphthalene tetrachloride and a 1% concentration of a proprietary dope, gave an increase of 60–70% over the base oil, being only slightly better than castor oil. The more powerful dopes, although unsuitable for use in engines, enabled loads between two and three times as great as the mean for the base oil to be carried.

In order to obtain some comparison between the gear-testing machine and the four-ball E.P. lubricant-testing machine, a few of the oils were tested in the latter machine at the National Physical Laboratory, and it is noteworthy that, with one exception, the correlation was fairly good, the oils being rated, qualitatively at least, in the same general order as the tests on the gear machines.

The second report summarises and concludes the investigation on the relative durability of a range of gear steels. The tests described are, in most respects, standardised, all being on the same size of gears (a 15/16 tooth ratio of 5 D.P.), lubricated with the same oil and run under the same conditions. The relative durability of the range of gear steels studied included case-hardening steels, direct-hardening steels, both cyanide hardened and without surface hardening, and one nitriding steel.

The case-hardened gears were found, in general, to withstand higher loads than direct-hardened gears, though no consistent difference was noticeable between the various case-hardened steels, except that the plain nickel alloy steels appeared to scuff more readily than the nickel-chrome steels when the profiles were imperfect.

The effect of profile form and modifications was studied in relating to scuffing and scoring, but the form did not appear to have any direct effect on the liability to pitting during prolonged running. The main factor affecting pitting appeared to be the surface hardness, since the direct-hardened gears, and any case-hardened gears which were rather low in hardness or thin in the case, gave early pitting.

Some of the case-hardened steels appeared to be more sensitive to slight differences in heat-treatment than others, in respect of the depth of hard case obtained, this being particularly noticeable if profiles had to be ground twice. The nitrided gears gave complete freedom from wear up to the load at which surface failure occurred, but at this load they became badly crushed and pitted in a manner suggesting that they would not be suitable for duty where overloads might be encountered.

The Behaviour of Steel at Low Temperatures when subjected to Tension-Compression-Fatigue Tests

FURTHER work on the behaviour of steel at low temperatures is reported by Mr. Hempel and J. Luce.* The subject is introduced by a detailed survey of published work on fatigue tests of steel at low temperatures and a description is given of the construction and operation of the testing machine used in the present investigations. Tests have been carried out on plain test-pieces as well as on those having a V-notch. The materials tested compressed two chrome-molybdenum steels, one chrome-nickel-molybdenum steel, and three carbon steels containing 0·08, 0·40 and 0·64% carbon, respectively. The tests were carried out at 20°, −78°, and −188° C., and at various mean pressures, using the Wöhler method.

The fluctuations of the temperature of the bath were about ± 2° and ± 3° C. at − 78° C. (alcohol and solid carbonic acid), and at − 188° C. (fluid air) respectively. The temperature was distributed equally along the gauge length of the specimens at − 188° C., but at − 78° C. there was an increase of the temperature of 3° at the ends against the middle of the gauge length. In some cases the changes of the temperature were considerably larger; they could be observed especially on solid specimens of two of the carbon steels shortly before the fatigue failure; the reason obviously being an insufficient cooling down during the fatigue tests under high load near the yield-point.

The Wöhler curves show that, generally, the various mean stresses have no influence on the course of these curves for a particular temperature: they are nearly parallel; but it is remarkable that the part of the Wöhler curve that is inclined to the abscessea-axis takes a different course with different temperatures and different shapes of the specimens. The fatigue resistance diagrams show nearly parallel limit lines for the solid specimens of the various steels also at low temperatures, and a small decrease of the deflections with increasing mean stress. Quite a different course of the limit lines of the stress were observed from notched specimens—i.e., a large decrease of the deflection with increasing mean stress.

Fatigue tests on solid and notched specimens at low temperatures can be continued to considerably higher mean stresses than at a temperature of + 20° C. The increase of the field between the limit stresses is largely influenced by the figure of the yield-point for the various temperatures. When comparing the fields of tensile strength and fatigue strength it can be stated that no fundamental change, but only an increase of the stress deflections, takes place in the course of the limit-stress curves, the point of intersection of the curves of the upper limit stress and of the yield-point being shifted to smaller mean stresses in the case of the static tensile strength.

Fatigue strength as well as notched fatigue strength increase with increased tensile strength of specimens. That is especially clear in the case of solid specimens at low temperatures of − 78° and − 188° C., but can also be observed to a smaller degree with notched specimens. The notch sensitivity of carbon steel increases considerably with decreasing temperature, especially at − 188° C.; that is clear by the decrease of the ratio of the fatigue strength of notched specimens to that of solid specimens.

Although there are some fundamental differences of behaviour of the carbon and alloyed steels with regard to notch-toughness and fatigue strength of notched specimens at low temperatures, the result of this investigation permits the conclusion that no decrease of fatigue strength takes place with decreasing temperature for solid as well as for notched specimens.

Generally, it must be added, that the experimental results are not sufficient to develop a law which would be valid for general application with regard to the influence of low temperatures on the fatigue strength of various qualities and shapes of steel specimens. However, the experiments show that figures of fatigue strength at low temperatures can be determined by the Wöhler method and by the usual testing machines with simple additional appliances, but the experimental method should be developed to ensure a high degree of accuracy; the damping capacity of the material at these temperatures should be determined, and the investigations should be continued to determine more definitely the increase of the fatigue strength as well as the different course of the Wöhler curves at temperatures from − 80° to − 150° C.

The practice of applying notch-toughness tests in addition to yield-point, tensile strength, and elongation, when selecting steel for stresses at low temperatures, for barrels, tanks and other vessels, gears, motors, etc., may be considered as correct according to these investigations, they also confirm the correctness of the present wide-spread use of carbon steels for machines and constructional parts exposed to low temperatures; but, of course, this kind of strain is always decisive for the final choice of the material. Attention is particularly drawn to the fact that a careful treatment of the surface is necessary if use is to be made of the high tensile strength of alloy steels for fatigue stress at low temperatures.

* Mitteilungen aus dem Kaiser-Wilhelm Institut für Eisenforschung zu Düsseldorf, vol. xxiii.

Electrolytic Behaviour of Ferrous and Non-Ferrous Metals in Sub-Corrosion Circuits

MEASUREMENTS have been made on the behaviour of a variety of metals exposed to corrosion in soils having widely different chemical and physical properties. The metals studied were low-carbon steel, two varieties of stainless steel, copper, low-copper brass, zinc and lead. A specially designed corrosion cell, previously used in studying the corrosion of steel, was employed. With the more corrodible metals, such as steel and zinc, and other metals in certain environments, the cell develops its own e.m.f. For measuring the corrosion rate of these metals for comparison with the results of field tests, the current required to bring the electrodes of the corrosion cell to the same potential was selected. For all other materials and environments in which the corrosion cell did not develop a definite polarity, the minimum current required to protect the cathode of the cell from corrosion was taken as equal to the corrosion current. The current corresponding to the equipotential value, *i.e.*, the corrosion potential, was found to be equal to the protective current when the rate of corrosion is determined by the reaction at the cathode.

The rates of corrosion of the metals in the different soils were in agreement with the results of the field tests to the extent that both series of tests indicated whether or not the materials were seriously corroded in the different soil environments. Zinc, however, which was corroded more rapidly than steel in the laboratory, was more resistant to corrosion than steel in the field tests.

The electrode at which the corrosion rate is controlled was determined for the different materials and environments by observing current potential curves of the separate electrodes. The rates of corrosion of steel and zinc under most soil conditions are controlled by the reaction at the cathode. However, in very porous soils, particularly those strongly alkaline in reaction, the control shifts to the anode. The corrosion of copper, brass and stainless steel, which in most soils is controlled anodically, is controlled by the cathode in poorly aerated organic soils which contain high concentrations of soluable salts, indicating that the normally protective oxide film has been destroyed. For lead, however, the shift of control is in the opposite direction, becoming anodic in the presence of soluble salts because of the formation of a protective film of corrosion products at the anode.

*J. A. Denison, The Electrochemical Society, General Meeting, April, 1942; reprint 81-87.

Analytical Control of Nickel-Plating Solutions

By E. E. Halls

Control of plating solutions is an essential factor in modern plating practice, and in this article the author treats the subject in a manner sufficiently comprehensively to embrace all types of solutions encountered, but by sectionalising the information an easy reference is presented for the more simple formulations.

ELECTROLYTES containing a mixture of single and double nickel salts (viz., nickel-sulphate and nickel-ammonium-sulphate) as the source of nickel ions, with either sodium or ammonium chloride present to promote anode corrosion, and boric acid as a buffer agent for purposes of reducing free acidity, represent the largest established nickel-plating solutions. Deposition has largely been effected in the cold. The exigencies of modern competitive mass production methods have demanded cheaper and quicker processes. As a result, the last decade has seen the gradual development of more heavy duty electrolytes, bearing a higher nickel content and operated warm, while a greater range of free acidity has been proved practicable for some purposes. Thus, the use of double salt has declined; nickel-chloride extensively replaces the ammonium or sodium salt as the source of chloride ions, while temperatures up to 60° C. are common, and even up to 70° C. occasionally encountered. Moreover, the popularity of zinc and aluminium base diecastings, and the need for nickel-plate finish upon them has necessitated the evolution of special electrolytes for their expeditious handling. The inclusion of sodium and magnesium sulphates in baths for the treatment of these alloys has resulted. The Bureau of Information on Nickel brochure, "American and Continental Practice in Nickel Deposition," reference L 4, gives perhaps the most comprehensive survey in a compact manner of the range of nickel-plating solutions popularly favoured. A study of this would, at first sight, suggest that the analytical control of nickel-plating solutions is likely to be a laborious task. In routine practice, however, this is not the case. A suitable bath formulation once decided upon for a specific class of work is adhered to, and the electrolyte is prepared and maintained from ingredients of known standard. The condition of the solution under these conditions can be ascertained by analysis for certain quantities at regular intervals and by complete analysis at only infrequent periods. The fundamental quantities with which the chemist is concerned are :—

1. Total nickel which has been introduced as one or more of the following :—
   - (*a*) Single nickel salts.... $NiSO_4\ 7H_2O$.
   - (*b*) Double nickel salts .. $NiSO_4\ (NH_4)_2SO_4\ 6H_2O$.
   - (*c*) Nickel chloride ..... $NiCl_2.\ 6H_2O$.
2. Total chlorides, added as one or more of the following :—
   - (*a*) Nickel chloride ..... $NiCl_2.\ 6H_2O$.
   - (*b*) Sodium chloride..... NaCl.
   - (*c*) Ammonium chloride .. $NH_4.\ Cl$.
3. Total sulphates, including that entering with the nickel salts and added as sodium $Na_2SO_4.10H_2O$ or magnesium $MgSO_4.7H_2O$ sulphate (when present).
4. Total ammonia content, entering as double salt or as chloride.
5. Boric-acid content. (H_3BO_3)
6. Free acidity (expressed in terms of sulphuric acid H_2SO_4, or as *p*H + value).

The tendency, nowadays, is to employ continuous filtration, employing a positive system whereby solution is pumped from the vat through filters and back to the vat, and by which means a crystal clear electrolyte is maintained. In such cases the solution direct from the vat without filtration may be analysed. If simple felt-bag gravity filters only are used the solution is not transparent, and it must be filtered before analysis; the first 20 c.c. of the filtrate should be rejected, while the sludge (mainly nickel hydroxide with some hydrated iron oxide) may be analysed if desired.

Stock Solution for Analysis.—As the volume of plating solution required for each test is small, it is usually deemed more accurate to take, say, 100 c.c. of plating solution (filtered if necessary), dilute to exactly 1 litre, and thoroughly mix. This applies to all tests except *p*H+ value, which, of course, must be determined on the actual electrolyte itself. For the sake of uniformity, therefore, it is assumed in the following that undiluted plating solution is used throughout.

Section 1.—Total Nickel Content

A volumetric method is very rapid, and with a little experience at spotting the exact end-point is exceedingly accurate. It is consequently always employed in routine work, but the gravimetric determination may periodically be desired for check purposes.

(*a*) *Volumetric Determination of Total Nickel Content.*—The following reagents are required :—

Exactly N/10 silver-nitrate solution, prepared from 16·99 grms. of pure silver nitrate per litre of distilled water.

Approximately 2% aqueous solution of potassium iodide.

Exactly N/10 sodium-cyanide solution, prepared from 10 grms. sodium cyanide per litre of distilled water, standardised against the silver nitrate and reduced to exactly N/10 for convenience. Alternatively, the cyanide solution may be labelled with the necessary normality factor. It should be noted, however, that if it is stored for long periods, periodic re-standardisation should be made.

The standardisation is carried out by running about 25 c.c. of the cyanide solution from a burette into a 350 c.c. Erlenmeyer flask, adding 5 c.c. potassium-iodide solution, and then running in silver nitrate from a second burette till a permanent precipitate is obtained. Cyanide is again gradually added until the precipitate just clears.

For the nickel determination, 1c.c. of the plating solution is pipetted into a 350 c.c. Erlenmeyer flask, about 20 c.c. water and 5 c.c. potassium-iodide solution added. From the silver-nitrate burette a little of the silver solution is added to give a definite opalescence, and then from the sodium-cyanide burette cyanide is run in gradually to clear the solution. Finally, the excess of cyanide is back titrated with the silver solution, the end point being taken as the first sign of permanent opalescence.

Note 1.—The 1c.c. of plating solution may be directly titrated with sodium cyanide until the precipitate which first forms just clears, but the above modification introducing the use of potassium iodide and silver nitrate is considered to render the reading of the end point much more exact.

Note 2.—When low pH+ nickel solutions are involved, that is, below pH+ 5, the 1c.c. of plating solution should be made just alkaline with dilute ammonia before titrating.

If (a) c.c. of N/10 sodium-cyanide solution and a total of (b) c.c. of N/10 silver-nitrate solution are employed in titrating the 1c.c. of plating solution, then :—

Total nickel content of plating solution = 0·469 (a—b) oz. per gal. or

Total nickel expressed as single salt, $NiSO_4.7H_2O$ = 2·25 (a—b) oz. per gal.

(b) *Gravimetric Determination of Total Nickel Content.*—The dimethylglyoxime method is employed, the reagent required being prepared by dissolving 15 grms. of dimethylglyoxime in a litre of 64·0 P. methylated spirit and filtering before use.

1 c.c. of the plating solution in a 600-c.c. beaker is diluted to 150—200 c.c. with distilled water and 2 grms. of tartaric acid in a little water is added. The solution is made just alkaline with ammonia and heated to 80° to 90° C. Excess (100 c.c. is usually ample) of dimethylglyoxime solution is added to the hot solution. The mixture is thoroughly stirred and allowed to stand, preferably in a warm place, for 15 mins. The red precipitate of nickel dimethylglyoxime is filtered on a prepared Gooch crucible or on a filter-paper, dried for 2 hours at 110° C. and weighed. It is washed thoroughly with a hot mixture of 50/50 water and industrial methylated spirit, and finally with hot water. After drying to constant weight at 110° C. (2 hours usually suffices), the crucible or filter-paper is reweighed.

If "c" grammes of nickel dimethylglyoxime is obtained from 1 c.c. of plating solution, then :—

Total nickel content of plating solution = 32·51 c oz. per gal. or

Total nickel expressed single salt, $NiSO_4.7.H_2O$ = 155·6 c oz. per gal.

Section 2,—Total Chloride Content

Again, this is most conveniently determined by a rapid volumetric process in routine work, with an occasional gravimetric check if desired.

(a) *Volumetric Determination of Total Chloride Content.*—2 c.c. of the plating solution is pipetted into a 350-c.c. Erlenmeyer flask and diluted to about 10 c.c. with distilled water. About five drops of 10% potassium chromate solution are added, and the mixture titrated carefully till a permanent reddish-brown colour develops.

Note 1.—If the plating solution is strongly acid, e.g., below pH+ 5·0, neutralisation with dilute ammonia prior to titration with silver nitrate is advocated.

Note 2.—If the plating solution has become markedly alkaline, e.g., above pH+ 7·5, neutralisation with dilute acetic acid is recommended.

If "d" c.c. of N/10 silver nitrate solution are required for the titration of 2 c.c. of plating solution, then :—

Total chloride content of plating solution expressed as Cl = 0·284 d oz. per gal. or

Total chloride expressed as nickel chloride $NiCl_2.6H_2O$ = 0·950 d oz. per gal. or

Total chloride expressed as sodium chloride NaCl = 0·468 d oz. per gal. or

Total Chloride expressed as ammonium chloride NH_4Cl = 0·428 d oz. per gal.

(b) *Gravimetric Determination of Total Chloride Content.*—2 c.c. of the plating solution is pipetted into a 400-c.c. beaker, slightly acidified with nitric acid and diluted to about 200 c.c. with distilled water. After heating to 80° to 90° C., chloride is precipitated by gradually adding silver-nitrate solution with constant stirring until a small excess is present. The precipitate of silver chloride is allowed to coagulate and then filtered on a prepared Gooch crucible. It is washed with water acidified with a few drops of nitric acid until the filtrate is free from silver salts, and finally with hot water till free from acid. The crucible is then dried at 100° C., and finally heated to constant weight at 130° C.

If "e" grammes of silver chloride are obtained from 2 c.c. of plating solution, then :—

Total chloride content of plating solution expressed as Cl = 19·79 e oz. per gal. or

Total chloride expressed as nickel chloride $NiCl_2.6H_2O$ = 66·2 e oz. per gal. or

Total chloride content expressed as sodium chloride NaCl = 32·6 e oz. per gal. or

Total chloride content expressed as ammonium chloride NH_4Cl = 29·8 e oz. per gal.

Section 3.—Total Sulphate Content

It is usually necessary to determine sulphates only when sodium or magnesium sulphates are present in the electrolyte as essential constituents, the sulphate radicle being more amenable to rapid estimation than is either sodium or magnesium.

1 c.c. of the plating solution is pipetted into a 600-c.c. beaker, diluted to 250/300 c.c. with distilled water. 3—5 c.c. of hydrochloric acid is added, and the solution heated short of boiling. 20 c.c. of 10% barium chloride is added with stirring, and the solution simmered for an hour. It may then be filtered with quite sufficient accuracy for the purpose, although for exactness it is preferably allowed to stand overnight. The precipitate of barium sulphate is thoroughly washed with hot water, and then ignited wet in a tarred silica crucible to constant weight.

If "f" grammes of barium sulphate are obtained from 1 c.c. of plating solution, then :—

Total sulphate content of plating solution expressed as single salt $NiSO_4.7H_2O$ = 193 f oz. per gal. or

Total sulphate content of plating solution expressed as double salt $NiSO_4.(NH_4)_2SO_4.6H_2O$ = 135 f oz. per gal. or

Total sulphate content expressed as sodium sulphate $Na_2SO_4.10H_2O$ = 221 f oz. per gal. or

Total sulphate content expressed as magnesium sulphate $MgSO_4.7H_2O$ = 169 f oz. per gal.

Section 4.—Total Ammonia Content

This must generally be estimated if knowledge of the condition of the solution with respect to the accumulation of ammonium salts is desired, and only in the case of the simpler formulations can this quantity be computed from the other analytical values. It is most rapidly determined by distillation. A measured volume of the plating solution is pipetted into a flask and the latter fitted with a tap funnel, splash-head and water condenser. An adaptor from the latter delivers below the surface of a suitable volume of N/2 hydrochloric acid contained in a 350-c.c. Erlenmeyer flask.

25 c.c. of plating solution in the flask and 25 c.c. N/2 HCl in the receiver are suitable volumes. An excess of 10% caustic soda is run in from the tap funnel, and the solution simmered till the whole of the ammonia is collected. Excess of hydrochloric acid is back titrated with N/2 sodium-hydroxide solution, using methyl orange as indicator. The difference between the volumes of N/2 hydrochloric acid and N/2 sodium hydroxide is the ammonia equivalent. If this volume is "g" c.c. working on 25 c.c. of plating solution, then :—

Total ammonia content of the plating solution expressed as NH_3 = 0·0544 g oz. per gal. or

Total ammonia content expressed as double salt $NiSO_4.(NH_4)_2SO_4.6H_2O$ = 0·632 g oz. per gal. or

Total ammonia content expressed as ammonium chloride NH_4Cl = 0·171 g oz. per gal.

Section 5.—Boric Acid Content

As this constituent is relied upon as a buffer to depress free acidity, data upon its concentration from time to time are important. The following is the most convenient rapid

test. The boric acid reagent is kept as a stock reagent and is prepared as under :—

60 grms. of sodium citrate are dissolved in 400 c.c. distilled water, 600 c.c. pure glycerol, and 10 c.c. of 2% alcoholic solution of phenolphthalein are added and thoroughly mixed. The mixture is titrated to a faint permanent pink with N/10 caustic soda.

To make the boric acid estimation, 1 c.c. of the plating solution is pipetted into a 350-c.c. Erlenmeyer flask. It is exactly neutralised by adding the requisite amount of acid as shown by the acid or alkali titration, section 6. (Note : One-fifth of the actual test volume is required, since 5 c.c. of plating solution is used for acidity or alkalinity test and only 1c.c. for the boric acid determination.) 25 c.c. of the boric acid reagent is then added and the mixture titrated with N/10 sodium hydroxide to an end point, revealed by a colour change from green to blue. If "h" c.c. of N/10 sodium hydroxide are required for 1 c.c. of plating solution, then :—

Boric acid content H_3BO_3 of plating solution $= 0{\cdot}99\,h$ oz. per gal.

Section 6.—Free Acidity (Acidity, Alkalinity or *p*H+ Value

Control to an exact free acidity value is the most critical factor in usual plating processes. Excessive acidity means loss of efficiency, gassing, porosity, etc., while a deficiency promotes sludging and weak spots in the deposited coatings due to inclusions.

(*a*) *Acidity or Alkalinity by Titration.*—This is convenient because it requires no special equipment or reagents, and it provides necessary information for the boric acid test (Section 5).

5 c.c. of the plating solution is measured into a 350 c.c. Erlenmeyer flask, and a few drops of 0·5% methyl-orange solution added. (A) If the latter indicates acidity (pink), the solution is titrated with N/10 sodium hydroxide till the pink changes to brown as end point. (B) If alkalinity is indicated (brown), the solution is titrated with N/10 hydrochloric acid to a pink colour as end point.

If "j" c.c. are used for titrating the 5 c.c. of plating solution, then :—

(A) Free acidity of plating solution expressed as $H_2SO_4 = 0{\cdot}157\,j$ oz. per gal. or

(B) Free alkalinity of plating solution expressed as $NaOH = 0{\cdot}128\,j$ oz. per gal.

(*b*) *pH+ Value.*—Up to a few years ago it was common practice to test for acidity from time to time during the working day with litmus-paper. Modern practice demands a more discerning method, and *p*H+ value provides this. Although *p*H+ value signifies the logarithm of the inverse of the hydrogen ion concentration, its determination by indicators involves very little effort more than the litmus test. A potentiometric method can be used if equipment is available, but greater skill is required. Either method serves the purpose of indicating whether the solution is within specified acidity limits, and whether sulphuric-acid additions are required to reduce alkalinity, or whether nickel-carbonate paste or ammonia should be introduced to correct excessive acidity. Both methods give reliable results capable of repetition, but they do not exactly coincide, so that the one type of test should be adopted and maintained.

A.—Determination of pH+ Value of Plating Solution by Indicator Method.—This comprises taking measured volumes of the plating solution and indicator, mixing, and comparing the resultant colour with standards. The indicator employed depends upon the *p*H+ value of the solution, and Table No. 1 shows a number of indicators, their useful *p*H+ range, and corresponding colour change. It is a wise procedure, as far as possible, to perform duplicate tests, using two indicators of overlapping range. These indicators are supplied in consistent quality by chemical houses, ready prepared in solution, neutralised to their half-way point. The standards can most conveniently be prepared by utilising buffer mixtures, marketed by the concern from which the indicators are procured. These buffer mixtures are usually offered in tubes in quantity for dissolving in 1 litre of water, and from curves provided with them, the amounts of standard acid or alkali required to give particular *p*H+ values over the range 2 to 12 are derived. The necessary range of standard *p*H+ value solutions, in increments of 0·2, can thus be prepared, and the colour standards produced by sealing 10 c.c. of each solution with 0·5 c.c. of indicator in glass tubes. The tubes employed should be similar to those intended for the actual *p*H+ value test, identical in wall thickness, diameter and colour. Glassware must be thoroughly clean, washed first in distilled water, and then with the liquid for which it is to be employed.

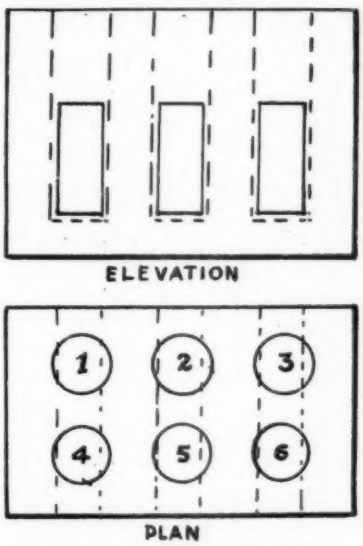


Fig. 1.—Comparator Block.

The actual test is performed on 10 c.c. of plating solution, rapidly cooled to room temperature, and 0·5 c.c. of indicator is added and thoroughly mixed. Colour comparison is facilitated by using a wooden block having two rows of three holes to take six of the test tubes, and three lateral slots for viewing in transmitted light. This is shown diagramatically in Fig. 1. The test sample is placed in position 2, with a tube of distilled water in front of it (position 5). Two standard indicator tubes, 0·2 *p*H+ unit apart, are placed in positions 4 and 6, with tubes of the nickel-plating solution behind them (positions 1 and 3). In this way the green colour of the plating solution is taken care of, and any masking effect that it would have upon the indicator colour eliminated. Standards in positions 4 and 6 are changed until either an exact match is found, or the colour of the test solution lies between the two standards.

TABLE I.

STANDARD INDICATORS FOR *p*H+ TEST.

Name of Indicator.	*p*H+ Range.		Colour Change.	
	From.	To.	From.	To.
Thymol Blue	1·2	2·8	Red	Yellow
Bromo-Phenol Blue	2·8	4·6	Yellow	Violet
Bromo-Cresol Green	3·6	5·2	Yellow	Blue
Methyl Red	4·2	6·3	Red	Yellow
Bromo-Cresol Purple	5·2	6·8	Yellow	Violet
Bromo-Thymol Blue	6·0	7·6	Yellow	Blue
Phenol Red	6·8	8·4	Yellow	Red
Cresol Red	7·2	8·8	Yellow	Violet Red
Thymol Blue	8·0	9·6	Yellow	Blue
Cresol Phthalein	8·2	9·8	Colourless	Violet Red
Alizarine Blue S.	9·0	11·0	Brown	Green
Alizarine Blue S.	9·0	13·0	Green	Violet

By carefully storing in a dark place when not actually in use the standards remain unchanged for several years. However, permanent coloured glass standards can be now obtained, dispensing with the work entailed in making the solutions.

B.—Determination of pH+ Value of Plating Solution by Electrometric Method.—Either the hydrogen electrode or the quinhydrone electrode may be employed. Using the former, the plating solution is placed in the electrode

vessel, the electrode (platinum freshly coated with platinum black) inserted, and a stream of hydrogen passed through the solution. The potential of the hydrogen electrode so produced is measured, when equilibrium is attained, in conjunction with a standard calomel half electrode on a suitable potentiometer arrangement, and computed to pH+ value.

The quinhydrone method is preferred because the use of hydrogen is eliminated and equilibrium is attained much more rapidly. A small quantity of the plating solution is transferred to the electrode vessel, a few crystals of quinhydrone added, and the electrode inserted. This electrode is a simple affair, a piece of polished platinum steel welded (oxy-hydrogen flame) to a platinum lead, cleaned on each occasion of use by heating to redness in an alcohol

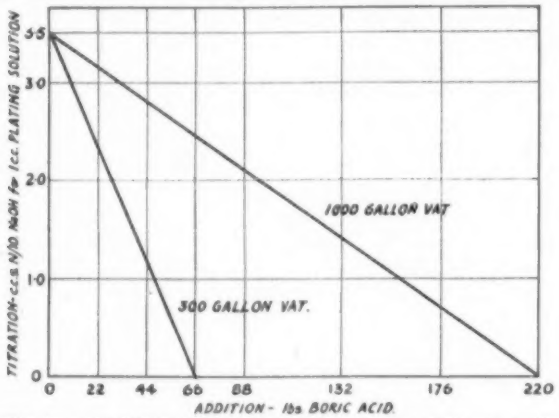


Fig. 2a.—Relation between test titration and boric acid additions; nominal 3½ oz. per gal.

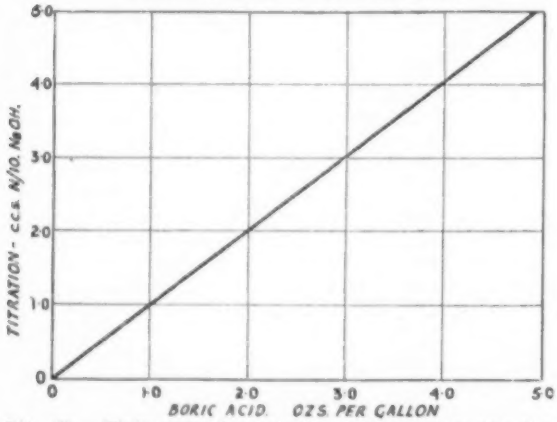


Fig. 2b.—Wall chart for relation between test titration and boric acid content per gallon.

blow-lamp. The potential of the single electrode is read in conjunction with a calomel half electrode on a potentiometer, and computed to pH+ value.

The potentials of the half electrodes produced by the plating solution depend upon their hydrogen ion concentrations, the relationship being linear. The equipment used can be checked against solutions of known pH+ value, and curves made for the easy conversion of test results. Those readers who desire detailed data on the subject are referred to Clark's "Determination of Hydrogen Ions."

Periodicity of Analysis

The frequency of analytical checks depends largely upon local conditions, including uniformity of work handled, strictness of supervision and general control of cleanliness, temperature and current density. In well-operated plants, pH+ value determinations twice daily, and nickel, boric acid and chloride analyses once a week suffice. This assumes an 8-hour working day; with 24 hours' working, the correspondingly more frequent checks are advocated. When sodium or magnesium sulphates are incorporated, or double nickel salts are present, they too should be evaluated fortnightly.

Computation of Results

The relationship between test results and ounces of constituent per gallon, as given under each section above, can be translated into simple straight line graphical charts. Or again, if all the vats are of the same capacity, they may be construed as pounds of constituent per vat, or even as pounds of constituent to be added to the solution.

The complex baths do not always permit the use of simple wall charts for translating analytical results to actual ounces of constituent per gallon, or to additions of constituent required in the vat. The following is a general procedure to cover all types of solution :—

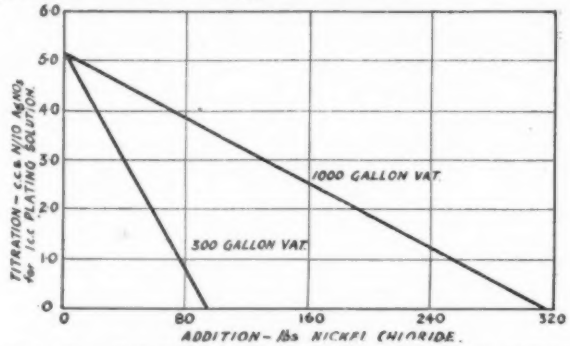


Fig. 3a.—Relation between test titration and nickel chloride additions; nominal 5 oz. per gal.

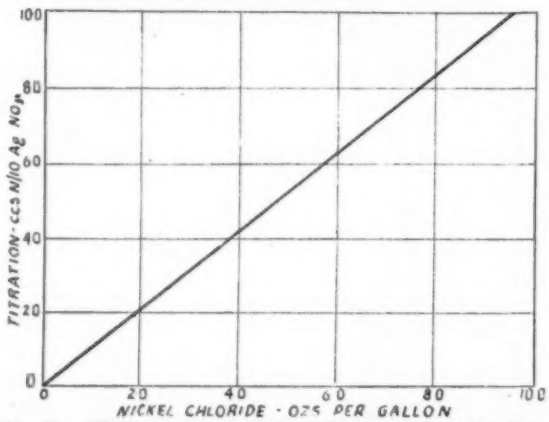


Fig. 3b.—Wall chart for relation between test titration and nickel chloride content per gallon.

1. *pH+ Value, Acidity or Alkalinity.*—One of these tests applies to all solutions, and from practical data compiled, curves can be drawn to show the quantity of addition necessary to correct a particular volume of plating solution.

2. *Boric Acid Content.*—This applies to all electrolytes. Fig. 2 shows a type of wall chart connecting analytical result with actual addition required to the plating vat, assuming a nominal 3·5 oz. of boric acid per gallon, while Fig. 2B shows graphically the connection between test result and the quantity of boric acid per gallon.

3. *Chlorides.*—Usually a simple chloride is present so that computation is simple, and Fig. 3A shows a typical type of wall chart from which to read of the necessary additions, assuming a nominal 5·0 oz. nickel chloride per gallon. Again, Fig. 3B merely connects the test titration value with chloride content per gallon of electrolyte. Similar conversion charts can apply to sodium or ammonium chlorides if one of these represents the chloride constituent.

4. *Double Nickel Salt.*—This is given by the ammonia estimation, Section 4, if double salts are the only source of ammonia.

If ammonium chloride is also present, the value determined by the chloride determination (Section 2) must be multiplied by 3·72 to convert it to the equivalent amount of nickel ammonium sulphate, and the result deducted from the nickel ammonium sulphate value derived from Section 4.

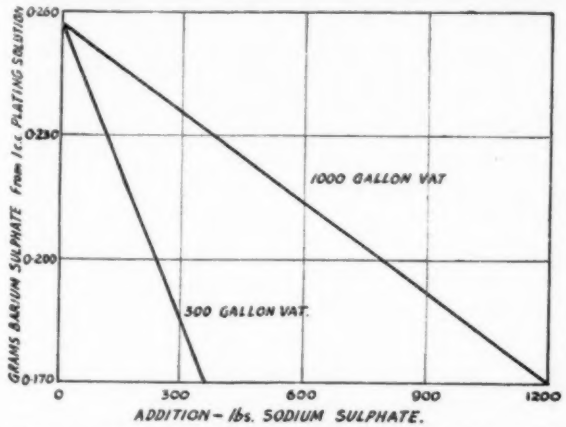


Fig. 4.—Sodium sulphate additions to vat from total sulphate test; nominal 23 oz. nickel sulphate, 19 oz. sodium sulphate.

5. *Nickel Sulphate, Single Salt.*—The results of Section 1 expressed total nickel content as single nickel salts, nickel sulphate. This represents the condition providing this is the only nickel salt present.

If nickel chloride is also a constituent, its equivalent nickel sulphate value, that is, 1·18 times the nickel chloride content found under Section 2 must be subtracted from the total nickel content expressed as nickel sulphate.

If nickel-ammonium sulphate is present, its value is oz./gal. multiplied by 0·71 must be deducted from the nickel-sulphate quantity.

6. *Sulphates.*—Sodium or magnesium sulphates are usually associated only with single salts.

The nickel-sulphate content (Section 1) can be deducted from the total sulphate expressed as nickel sulphate (Section 3), and the result multiplied by 1·15 to convert it to sodium sulphate, or by 0·88 to convert it to magnesium sulphate, oz./gal.

Alternatively, in practice it may be more convenient to utilise the total sulphate value expressed as sodium or magnesium sulphate, as the case may be, in conjunction with a wall chart from which to read additions of the sulphate to a vat of a particular capacity.

For example, for a solution of nominal formulation: single nickel salts, 23 oz.; boric acid, 3·5 oz.; nickel chloride, 2·5 oz.; sodium sulphate, 19 oz.; water, 1 gal: the nominal total sulphate expressed as sodium sulphate is 45·45 oz./gal., and Fig. 4 shows graphically the relationship between test result and sodium-sulphate addition required. This assumes, of course, that the nickel sulphates are already at correct concentration.

In the above treatment of the subject an endeavour has been made to make it sufficiently comprehensive to cater for all types of solution encountered, but at the same time to sectionalise it for easy reference for the more simple formulations.

Low Tin Solders

CONSERVATION of tin is of primary importance at the present time, so that a preliminary report by S. Turkus and A. A. Smith† of a series of tests on low-tin solders containing silver and bismuth is of interest. The addition of tin and bismuth to the lead-silver alloys overcomes certain disadvantageous properties of these alloys and at the same time retains some advantageous ones, and it is felt as a result of the initial tests that such solders do constitute very acceptable substitutes for high-tin solders in a great many cases.

Lead-silver solders have been known for many years, but because of their relatively high-melting temperatures and poor spreading properties they have not been widely used. The ternary alloys of lead-silver-tin, however, offer a variety of solders which have somewhat higher melting points than the 40–60 tin-lead solder, together with relatively good-spreading and bond-strength properties. Silver added to low-tin solders also lowers the liquidus temperature from 6° to 8° C. and appreciably improves the spreading properties, with but minor effect on the bond strength. One outstanding improvement of the silver-bearing solders is the increase in creep resistance. In Table herewith are some of the properties which have been determined for low-tin solders containing silver and bismuth, together with the properties of standard tin-lead solders for comparison. Careful standardised technique was used for all the tests carried out.

Alloys containing 15% tin, 5% bismuth, 1.5% silver, 1% antimony, and the balance lead, or 20% tin, 3% bismuth, 1.5% silver, 0.5% antimony, and the balance lead, are found to have liquidus temperatures slightly higher than that of the 40–60 tin-lead solder, together with a markedly higher tensile strength and a bond strength of approximately 80% of that of the binary alloy. The spreading properties are less than for the 40–60 tin-lead solder, but much greater than those of the binary lead-silver solders. It is considered, however, that, except for a few critical uses, this decrease in spreading properties is not of importance in the making of sound and reliable joints. By appropriate adjustment of the tin, bismuth and silver contents, fairly wide ranges of melting points and other properties may be obtained.

PROPERTIES OF SOFT SOLDERS.

Composition %.					Liquidus, °C.	Solidus, °C.	Tensile Strength Lb. per sq. in.	Bond Strength of Lapped Joints, Lb. per sq. in.	Spread of 0·5 grm. in sq. in.	Creep Rate in % per Year at 30° C.			
PB.	SN.	AG.	BI.	SB.						200 lb. per sq. in.	400 lb. per sq. in.	600 lb. per sq. in.	800 lb. per sq. in.
60·0	40·0	—	—	—	238	183	5,660	6,270	1·30	10·9	92·0	300 0	—
97·5	—	2·5	—	—	304	304	4,980	3,740	0·19	0·1	0·15	—	0·50
95·0	—	5·0	—	—	375	304	4,915	4,340	0·20	0·18	0·22	—	0·45
90·0	10·0	—	—	—	298	183	4,850	4,960	0·27	2·5	12·0	—	—
87·85	10·0	2·25	—	—	290	—	4,950	5,000	0·41	—	2·3	8·0	—
80·0	20·0	—	—	—	275	183	4,940	5,680	0·37	6·7	18·7	—	—
78·0	20·0	2·0	—	—	267	—	5,620	5,550	0·57	—	—	—	—
70·0	30·0	—	—	—	257	183	5,390	5,770	0·83	10·0	50·0	—	—
69·0	30·0	1·0	—	—	251	—	8,810	5,620	0·83	—	40·0	20·0	—
78·5	15·0	1·5	5·0	—	264	—	4,960	5,310	0·47	—	—	—	—
77·5	15·0	1·5	5·0	1·0	258	—	8,000	5,090	0·29	—	—	—	—
74·85	20·0	1·5	3·0	0·5	258	—	8,120	5,380	0·39	—	—	—	—

NOTE: Bond and spread tests were made on copper sheet.
Tensile and creep tests were made on chill-cast strips of the alloys.

Commercial trials and rapid hand soldering have been made on some of these alloys with excellent results, and automatic machine soldering has also been done with satisfactory results. Tin cans made on automatic machines have passed all the standard tests for 40–60 and 50–50 tin-lead solders. It is therefore considered that a more general use of such solders would effect a substantial saving in tin consumption, and as silver and bismuth are available in reasonable quantities it is probable that ample supplies for such alloys could be obtained.

† *Metals and Alloys*, 1942, Vol. 15, No. 3, pp. 412–413.

Treatment of Large Deposits of Low-Grade Chromite Ores

Under the strategic metal programme inaugurated by the United States Government, considerable attention is being directed to the development of domestic chromite deposits and to their utilisation in the war effort. Much progress has been achieved and a brief outline of the position is given.

A NEW metallurgical process for the treatment of large American deposits of low-grade chromite ores has been developed by the Bureau of Mines. Introduced as a result of many years of research and experimentation, this new process has successfully passed all tests made so far and has proved to merit large commercial production. Definite recommendations for the construction of commercial plants will be made when final data are received on additional tests in progress. A specific production programme is under consideration.

The new method, known as a roasting and leaching process, is designed to convert chromite concentrates into a higher grade of material which may be used to produce either high purity chromium or standard ferro-chromium for the manufacture of alloy steels. The process was developed in the Bureau of Mines Laboratories at Boulder City, Nevada, and Salt Lake City, Utah, under the direction of Dr. R. S. Dean. It is stated to be capable not only of increasing the chrome content in the concentrates, but also of raising the ratio of chromium to iron from about 1·7 to 1 up to as much as 30 or 40 to 1.

Vast deposits of low-grade chromite reserve ores in Montana, estimated at two and a half million tons, and deposits of similar ore in other western States are expected to furnish the raw materials for concentrated plants and proposed processing plants. The Montana deposits are believed to contain enough chromium to yield over 900,000 tons of chromite (concentrates containing 45% or more chromic oxide) of a grade suitable for making ferro-chromium.

It is well known that chromium is one of the most important of the elements which have contributed to the development of alloy steels. Chromium, added to steel in various proportions, either alone or in combination with other alloying elements, imparts special qualities of hardness, toughness, strength and resistance to wear or corrosion. It is invaluable in the manufacture of armour plate and armour piercing shells; chromium is also used in the manufacture of some high-speed tool steels and in many other vital war applications, such as for bearing surfaces, gauges, files, and in electrical heating elements. Stainless steel, which is also contributing considerably in the present emergency, is another important application for chromium.

While the largest industrial use of chromium is in the metallurgical field, more especially in alloy steel manufacture, it is highly important as a refractory in furnace construction. In some cases the entire furnace hearth may be built of chromite, but the more common application is as an insulating layer between hearth and roof in a basic furnace, since the chromite is neutral in composition and will not react with hearth or roof. Chromite also goes into a number of chemicals for industrial uses, many of which are important to military supplies, such as the tanning of leather. Dyeing, bleaching oils, paint pigments, and the treatment of boiler feed water all employ certain chromium chemicals. It is probable that the metallurgical and refractory industries absorb about 85% of the chromite consumption, the remainder being used by the chemical and paint industries.

In 1940 the United States consumed more than 600,000 tons of chromite, and, with the exception of 2,662 tons mined in that country, all was imported from abroad. It was shipped from Africa, the Philippines, India, New Caledonia, Turkey, Cuba, and Greece. During the first nine months of 1941 imports reached approximately 650,000 tons, while that obtained from ore mined in the United States rose to about 13,000 tons—still a very small part of the total consumption. With increasing wartime demands, it may be expected that chromite consumption will advance to a new high level this year and in 1943.

The Philippines, which in 1940 supplied the United States with 156,000 tons of chromite, have since been isolated by the war in the Pacific, and the 160,000 tons received annually from India, New Caledonia, Greece and Turkey is now in the uncertain category due to the war and shipping conditions. For these reasons the Bureau of Mines and the Geological Survey of the Interior Department co-operated in exploring a number of known chromite deposits in the United States. This work indicated that the deposits in Stillwater County, Montana, were by far the most important source; estimates indicate that in this section the reserves total over two and a half million tons. These chromite ores, however, are of a lower grade than the ores normally imported by the United States for making ferro-chromium used in steel alloys, for not only are they relatively low in chromic oxide (Cr_2O_3) content, but the ratio of chromium to iron in the ores is relatively low, ranging from 1·6 to 1·8 to 1, with an average of 1·7 to 1. For the production of standard ferro-chromium for the steel industry, it is not only necessary to bring up the chromic oxide content of the ore, but the ratio of iron should be at least 3 to 1.

The application of the low-grade chromite ores of Montana has been a problem of interest to the Bureau of Mines for some years; it has been attacked in two ways: (1) To develop a process for producing pure chromium metal by electrolytic methods; and (2) to produce by metallurgical operation a product which might be used for making standard ferro-chromium. This work has been quite successful along both lines, but the greatest immediate importance at present is the process for producing a concentrate that is readily usable for making standard ferro-chrome. Removal of an excess of iron from the chromite ores is the chief problem, and experiments have shown that this can best be done by the reducing roast and leaching process. In this method, after the chrome ore concentrates are received from mining and concentration plants, they are mixed with coke and treated in a specially designed rotary kiln. The material thus treated is cooled, and part of the iron is removed by gravity concentration or by magnetic methods. Further reduction of the iron control may be obtained by leaching with sulphuric acid, or sulphur dioxide, leaving the residue enriched in chromium. This final residue may contain about 30 times as much chromium as iron, but the results of research indicate that it probably will be most economical to produce a residue having a ratio of 5 parts chromium to 1 part iron. This will permit mixing of 2 tons of residue with 1 ton of raw concentrates to produce a product having a chromium-iron ratio of 3 to 1. This product may then be reduced directly by electric furnaces to produce standard ferro-chrome containing about 70% chromium.

The Effect of Zinc, Metal, Lead and Tin on the Properties of L.3 Alloy Sheet

The annealing, heat-treatment and room temperature ageing characteristics of alloys of normal L.3 composition, containing one of the elements zinc, nickel, lead and tin, have been investigated by Dr. M. Cook and R. Chadwick, as well as the mechanical and corrosion-resisting properties after heat-treatment at 495° C., followed by room-temperature ageing. A brief résumé of the investigation is given below.*

IN the commercial production of alloys impurities are introduced through the use of scrap and secondary metal. The nature and extent of these impurities naturally vary, and in the production of the aluminium alloy L.3 those most likely to be introduced in this way are iron, zinc, nickel, lead and tin. In order to determine the effect of these elements on the mechanical properties, age-hardening characteristics and corrosion resistance of the alloy an investigation has been made on four series of alloys of normal L.3 composition, each with progressively increasing proportions of one of the elements zinc, nickel, lead and tin, prepared in sheet form. The composition of the alloys investigated are given in Table I.

TABLE I.
COMPOSITION OF ALLOYS.

	By Chemical Analysis.					By Spectrographic Analysis.				
%	Copper, %	Magnesium %	Manganese, %	Silicon, %	Iron, %	Zinc, %	Nickel, %	Lead, %	Tin, %	Titanium, %
Zinc Series.										
Zinc.										
0·005	4·07	0·74	0·61	0·30	0·54	—	<0·005	<0·005	<0·005	trace
0·015	3·98	0·70	0·63	0·30	0·54	—	,,	,,	,,	,,
0·04	3·97	0·65	0·62	0·29	0·56	—	,,	,,	,,	,,
0·24	4·05	0·78	0·61	0·28	0·54	—	,,	,,	,,	,,
0·96	4·08	0·65	0·61	0·30	0·56	—	,,	,,	,,	,,
Nickel Series.										
Nickel.										
0·001	4·49	0·77	0·60	0·23	0·21	abt. 0·01	—	<0·005	<0·005	trace
0·008	4·35	0·75	0·60	0·24	0·24	,,	—	,,	,,	,,
0·045	4·33	0·78	0·60	0·24	0·21	,,	—	,,	,,	,,
0·20	4·38	0·77	0·60	0·22	0·21	,,	—	,,	,,	,,
0·40	4·25	0·77	0·60	0·22	0·21	,,	—	,,	,,	,,
0·80	4·27	0·80	0·59	0·21	0·21	,,	—	,,	,,	,,
Lead Series.										
Lead.										
0·002	4·40	0·79	0·60	0·15	0·19	<0·01	nil	—	<0·005	trace
0·13	4·47	0·77	0·61	0·13	0·19	,,	,,	—	,,	,,
0·28	4·48	0·73	0·60	0·14	0·16	,,	,,	—	,,	,,
0·48	4·42	0·72	0·60	0·14	0·20	,,	,,	—	,,	,,
Tin Series.										
Tin.										
0·002	4·13	0·73	0·62	0·27	0·29	0,01–0,02	<0·002	<0·005	—	trace
0·026	4·10	0·73	0·61	0·25	0·30	,,	,,	,,	—	,,
0·049	4·04	0·70	0·62	0·26	0·33	,,	,,	,,	—	,,
0·11	4·02	0·72	0·61	0·25	0·33	,,	,,	,,	—	,,
0·31	3·97	0·68	0·61	0·25	0·39	,,	,,	,,	—	,,

After casting and rolling the properties of the alloys were examined very carefully, but only those of greatest general interest are discussed. All the values are plotted in the form of curves, for in alloys of this type absolute values vary slightly from batch to batch, and therefore more useful information is derived from noting the trend of a curve than by a comparison of values. In each series, however, individual values plotted for the L.3 alloy without added impurity are typical of those normally obtained in commercially produced alloys.

The curve which best illustrates the characteristics of heat-treatable light alloys of the L.3 type is that represented by the solid line in Fig 1, of hardness after heating to various temperatures, followed by cold-water quenching. As the temperature of annealing rises the hardness values, both immediately after quenching and after subsequent ageing, increase rapidly up to 500° C., which is within a few degrees of the first melting of eutectic forming constituents.

Curves such as those shown in Fig. 1 were determined for each of the alloys in all four series, and none showed any variation in hardness, within the limits of experimental error, from the solid curves shown in Fig. 1, with the exception of the alloys containing nickel in excess of 0·2%. From those alloys differences from the standard values were apparent after quenching from temperatures higher than 350° C., the nickel causing a lowered hardness as measured either immediately after quenching or after the full ageing period of five days. The hardness values for the two alloys of highest nickel content are shown as broken lines in Fig. 1.

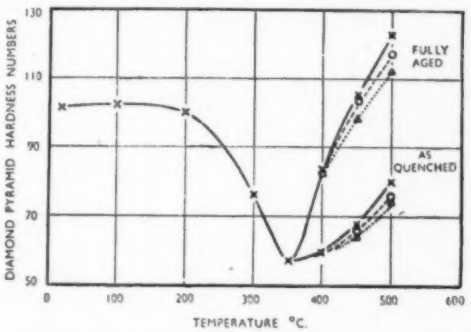


Fig. 1.—Annealing of hard-rolled sheet.

Observations on the age-hardening of alloys heat-treated at 495° C. over a period from the time of quenching up to 30 days. Again, no measurable differences were found for any of the alloys, from the normal curves for the alloy without added impurity, as shown in Fig. 2, in which logarithmic plotting for the ageing periods is used. For those alloys containing nickel, the age-hardening curves were of the same form, the hardness being lower throughout the ageing period to an extent depending on the nickel content, as shown by broken curves in Fig. 2. In all alloys age-hardening appeared to be virtually complete within five days.

In the heat-treated and fully-aged condition, D.P. hardness numbers, maximum stress, 0·1% proof stress, and elongation were determined on every alloy and the values are plotted in Fig. 3. each being the mean of three or more determinations. Tensile test-pieces were of ½ in. width and 2-in. gauge length, and heat-treatment was carried out on them after milling out from the sheet.

Of all the impurity additions made, again only nickel had any notable influence, and with this addition maximum stress, proof stress and elongation values were all lowered to an extent approximately proportional to the nickel content. Other additions were without influence on maximum stress or proof stress, although there was an indication of a general very slight lowering in elongation with increase in each impurity, but the evidence was hardly sufficient to consider such differences as established.

* *Journ. Inst. Metals*, May, 1942, pp. 169–178.

For the estimation of ductility, a bend test through 180° over a radius block was used. Tests were made immediately after quenching from 495° C., and also after annealing at 360° C., these being the two physical conditions in which bending is normally carried out commercially. The values plotted in Fig. 4 are limiting radius of bend (R) over thickness (T), the value R/T being zero where the alloy bends dead flat, whilst increasing R/T values indicate reduced ductility. Tested immediately after heat-treatment, most of the alloys gave an R/T value of about 1, the only notable differences being the alloys containing nickel, which showed a progressive diminution of ductility with increasing nickel content. In the annealed condition most of the alloys bent dead flat, so that it was impossible to ascertain differences in ductility, except in the case of the tin-containing alloys, where a slight increase in the R/T value with increasing tin content was noted.

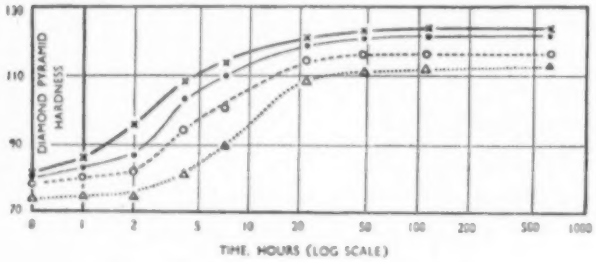


Fig. 2.—Age-hardening of heat-treated sheet.

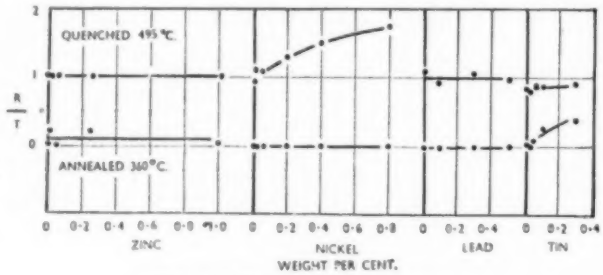


Fig. 4.—Results of bend tests.

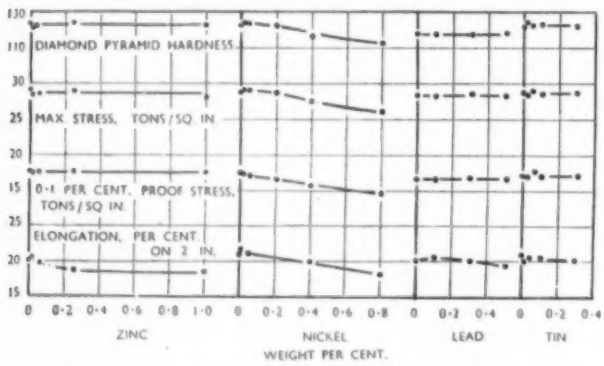


Fig. 3.—Properties of sheet quenched from 495 C. and fully aged.

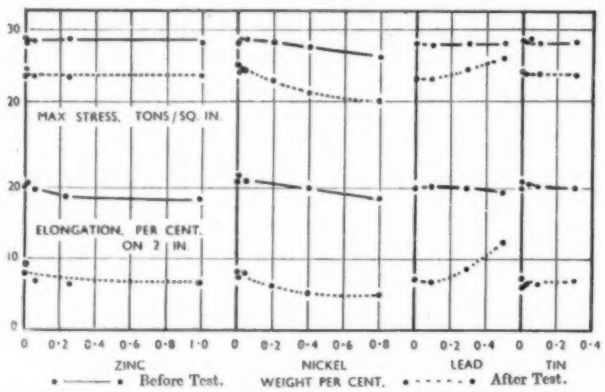


Fig. 5.—Physical properties before and after salt-spray test.

Experiments on the corrosion-resisting properties of the materials were carried out after heat-treatment at 495° C., followed by room-temperature ageing, for the alloy is invariably given this treatment before use, and it is now generally accepted that without such a final heat-treatment, combined with effective quenching, it is liable to severe intercrystalline attack on exposure to corrosive conditions. Test-pieces were exposed in a standard salt-spray cabinet, using a 3% aqueous sodium-chloride spray, for a period of two months. The spray was run during 8 hrs. each day only, and turned off during the night, during which period the specimens partially dried.

All samples were subsequently examined visually, and although they showed some surface pitting, there were no apparent differences between the surface appearance of any of them, with the exception of the two alloys of the higher nickel content, where attack appeared somewhat greater. On cross-sections of corroded test-pieces submitted to microscopic examination no instance of intercrystalline penetration was observed, and surface pits were all of a normal type. The results of tensile tests on the samples after a salt-spray test are shown in Fig. 5. Only two alloying additions appeared to influence in any way loss in strength by exposure to the salt spray, these being nickel, which had a slightly unfavourable effect, and lead, which had a definitely favourable influence in the higher concentrations.

In order to test more thoroughly the freedom from susceptibility to intercrystalline attack, all the alloys were also submitted to an immersion test for two days in a 3% salt solution containing 1%, as (HC) of hydrochloric acid. This test is an extremely searching one for susceptibility to intercrystalline attack, but in none of the alloys was any such susceptibility found, all showed no more serious attack than pitting similar to that produced by the salt-spray attack.

While the results of these experiments indicate that zinc, lead and tin in small amounts are not deleterious impurities in respect of the characteristics and properties of the alloy, which have been considered in this investigation, it would seem that the maximum quantity of nickel which can be present, with an iron content of about 0·2%, without adverse effect, is about 0·4%. As the effect of nickel is similar to that of iron, the extent to which either can be tolerated is proably dependent upon the amount of the other which is present.

Sir Cecil Weir's New Appointment

AT the request of the Minister of Supply, Sir Cecil Weir, K.B.E., M.C., D.L., has been released by the President of the Board of Trade in order to take up the appointment of Director-General of Equipment and Stores in the Ministry of Supply in succession to the late Sir William T. Wilson. Sir Cecil Weir, who was Controller-General of Factory and Storage Premises at the Board of Trade, will retain his membership of the Industrial and Export Council.

The President of the Board of Trade has appointed Mr. P. A. Warter, the Deputy Controller General, to be Controller-General of Factory and Storage Premises in succession to Sir Cecil Weir.

The Chemical and Physico-Chemical Analysis of Iron and Steel

Twelve Years' Advancement

By E. C. Pigott

In this and some subsequent articles the author gives an exhaustive and descriptive bibliography of contributions on ferrous analysis published during the period 1930-41 inclusive. They reveal the developments in analytical methods which have become more direct and of wider application in meeting demands for speed combined with accuracy.

Introduction

Results of Research.—The wealth of specific information contributed to the chemical journals could profitably be exploited by the analyst on an infinitely larger scale. In a consideration of the reasons for the frequent prejudice against the adoption of new methods it is evident that in too many laboratories there is insufficient time or incentive for examining new processes at the bench. In regard to the papers themselves, academic considerations are observed at the expense of actual usefulness, authors being apt, for instance, to overlook the importance of investigating the influence of allied constituents, a common and serious defect which discourages interest and adoption whilst all too seldom do the new methods possess simplicity, specificity and rapidity, which form a threefold criterion of value.

Notwithstanding the invaluable abstract summaries, it cannot be denied that one of the principal handicaps is the lack of rapid translation facilities, depriving most chemists of an enormous fund of current information.

A visionary viewpoint sometimes apparent is that a mere perusal of the periodicals is adequate, little effort being made to utilise the actual processes offered. Once having prepared a complete set of routine methods the compiler may feel disinclined to make alterations or additions, but the growing practice of detailing at least one chemist to the confirmation of journal methods suggests that there are many exceptions to such indifference.

Edison once confided, " Before starting an experiment, I always read everything available on the subject and on related matters," and within the limits often imposed by a flood of literature, this policy is equally commendable to-day.

The Rise of Documentation.—The most ancient documents in chemistry are two remarkable Greek papyri believed to have been written in the third century. Though now of purely historical value, many records of experiments appeared prior to the *Transmutation Period* (1500–1650) when various textbooks or treatises on metallurgy and chemistry became available. The first scientific periodical the Royal Society's *Philosophical Transactions*, was launched in 1664 and has been issued continuously. The medicinal monopoly of chemical knowledge gave place to the *Phlogiston Period* (1650–1775), in which the search was one for rational causes, and following Lavoisier's discoveries, Crell published, in 1778, his *Chemisches Journal*, the earliest of all classical periodicals.

During the subsequent *Quantitative Period*, specialised journals have appeared in abundance, and to-day over 3,000 different periodicals are recognised by Chemical Abstracts. According to a 1936 list of contributions, 22·2% appeared in the U.S.A.; 18·1% in Great Britain; and 14·9% in Germany. Russia subscribed a percentage of 7·3; France 6·2; Japan 5·5; Italy 4·8; China 1·9; Sweden, 1·7; and Belgium, 1·6. Thus, English is by far the most important language scientifically.*

Foremost amongst actual analytical journals are the following :—

Analyst (1876).
Ind. Eng. Chem. Anal. ed. (1929).
J. Soc. Chem. Industry (1882).
Ann. Chim. anal. chim. appl. (1896).
Ann. falsi (1908).
J. Asso. Official Agri. Chem. (1915).
Mikrochem (1923).
Spectro. chim. Acta. (1939).
Z. anal. Chem. (1862).

British Chemical Abstracts (Applied Chemistry) were first published in 1882.

Research Tendencies

Outstanding in the advances made in ferrous analysis is the definite replacement of several " classical " methods by processes of recent introduction. The thousand or so published papers dealing with analytical research conducted within this period reveal, however, much re-arrangement of established sequences of reactions as well as a considerable amount of repetitive overlapping.

The true criterion of progress in this field of chemistry is the improvement of methods rather than the mere re-arrangement of minor details, which seldom affords any real advantage. Yet it is clear that fundamental reactions are now so satisfactorily understood and exploited, that pure originality is scarcely achievable. The increasing complexity of alloy steels and irons has created a need for specificity and considerable attention has been paid to obviating interference from the numerous elements now employed. Satisfactory methods have been evolved to meet several new alloy-additions, including columbium, selenium, lead, silver and zirconium. Throughout the entire field, refinement of detail is abundantly in evidence.

Photometric and Spectrographic Analysis

Absolute measurement of colour intensity, afforded by the photo-electric absorptiometer, has given an impetus to the development of colorimetric processes. Some difficulty lay in obtaining coloured compounds stable to the actinic rays. An important advantage is the measurement of colorations for which visual comparison solutions are not producible by mere subsequent additions of solutions of the constituent element.

In metallurgical analysis, there is a strong if sluggish tendency for chemical methods to be supplemented by rapid physico-chemical processes, and, significantly, this indicates that for finding composition, fundamental atomic properties serve better than chemical affinity. In this new sphere, spectrographic technique is of foremost importance. There is little likelihood of chemical analysis being rendered obsolete by such advances as the initial outlay for apparatus is considerable, and this technique provides itself with new problems. The tendency is rather for spectrographic analysis and chemical technique to be employed in conjunction, the former having important qualitative and confirmatory values.

* Since 1913 the German output of chemical papers has fallen from 34·4% of the world's total, to 18·7%. The British Empire and the United States now subscribe over 35%.

Where the scale of routine analysis is large, spectrographic technique has already replaced chemical methods for most constituents of ferrous metals, but in so far as the elucidation of the state of combination is concerned, the spectrograph may offer little competition, as the precise condition is due to chemical affinity. A minor issue in favour of true chemical analysis is that of the adequacy of "average" samples in cases typified by a bed of pig iron or a heap of scrap metal; tested spectrographically, many separate analyses would be required. There is little doubt, however, that even within the next few years this instrument will be applied on a revolutionary scale.

The Value of Precision

In general, it may be claimed that the reproducible accuracy now attainable by the chemist exceeds that required by present standards of production, and that in view of this precision the interpretation of analyses could be greatly extended. That many firms are still too technically conservative to avail themselves of analytical developments is largely due to the older procedures being suitable for unqualified staffs. Where such obsolete methods are favoured the results are relative rather than absolute, and recent alloy additions—with the consequent complexity of scrap-base—have rendered many of these processes quite untrustworthy. Despite this, both fantastic reliability and rapidity are often expected from such unscientific technique and primitive equipment. Notwithstanding the tremendous importance of simplicity in a method, the process should first be made specific for the element concerned.

The Place of Metallurgical Analysis

Those impressed by the cultural value of chemistry may feel that economic considerations of utility and efficiency are overstressed, yet obviously, the more rapid scientific control becomes, the greater benefits will accrue. It has been said that whilst pure chemistry reveals the unessential applied chemistry actually does provide practical needs. This may be far too sharp a distinction, yet it is of interest that analytical chemistry functions as a reciprocal link, the analyst utilising and supporting both sections. In terms of utility and breadth, chemical analysis is no less important than inorganic, organic or physical chemistry, for in any branch a knowledge of composition is a primary factor. Nor is there a more vital aspect of applied chemistry than the science of metals, and viewed in such perspective, metallurgical analysis is seen to occupy a very solid position, especially in manufacturing progress.

It is becoming increasingly clear that in the immense pattern of scientific control, metallurgical analysis merits far greater recognition and support. Closer collaboration on the part of engineer, metallurgist and chemist would doubtless advance the standard of production far beyond present conceptions. Indeed, only by fully availing itself of the results of scientific research can the tremendous democratic effort now launched succeed in safeguarding our national privileges.

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[Part II. on the determination of single constituents will be commenced in the next issue.]

A Rotation Induction Furnace

A rotating liquid crucible has been developed which permits contact between slag and metal, but prevents the molten slag coming into contact with the crucible. Slag-metal reactions have been studied with this type of furnace, and several features of experimental technique are described.

QUALITY control in the manufacture of many metals, and particularly steel, is closely associated with control of the slag in contact with the liquid metal in the furnace. The contamination of molten slag by crucible refractories has been a major difficulty in laboratory studies of slag-metal reactions, particularly in the case of oxidised slags used at the comparatively high temperatures of the basic open-hearth furnace. During recent years, a rotating liquid crucible which permits contact between slag and metal, but prevents the molten slag from coming in contact with the crucible refractory, has been developed,[1] and although this type of furnace presents some operational problems it has provided a promising means of studying slag-metal reactions. A study of such reactions during the last three years has been carried out, in such an improved type of furnace, in which several features of experimental technique have been developed ; and the furnace used, the methods employed for forming special refractories, adding the charge of slag and removing it at the end of the test, have recently been described and discussed by F. W. Scott and T. L. Joseph.[2]

If a container holding a liquid is rotated about its vertical axis, the inner surface of the liquid assumes the shape of a paraboloid under the simultaneous action of a centrifugal force and gravity. The internal dimensions of the rotating liquid crucible thus formed are dependent upon the diameter of the container and its speed of rotation. By applying the principle to molten metal, it is possible to form a paraboloid of liquid steel that will act as a crucible in which slag may be introduced and melted. Due to the difference between the surface tension of molten steel and molten slag, the slag will remain within the cup of metal and will not come in contact with the crucible wall. In this way, the contamination of the slag by the crucible refractory is avoided. With such a crucible, its rotating base and the coils of a high-frequency induction furnace are enclosed in a gas-tight box, the furnace atmosphere can be controlled and with high-frequency induction heating the temperature can also be controlled within narrow limits. Control of temperature, slag and metal composition, and the gas phase make equilibria studies of slag-metal reactions feasible.

In the slag-metal reactions discussed, the apparatus consisted of an alundum crucible fitted into a graphite sleeve standing on a magnesium spindle. This spindle was bolted on to a cast-iron plate rotated by means of a shaft passing through the top of a table on which were mounted the inductor coil and the gas-tight box surrounding the crucible. The inductor coil consisted of 35 turns of $\frac{3}{8}$ in. flattened copper tubing separated from each other by $1\frac{1}{16}$ in. layers of sheet mica with an inorganic binder, and was mounted on one side of a box 10 in. by 10 in. and 12 in. high fastened to the table. Electrical leads and cooling water connections were provided on the outside of the box. The height of the box directly over the top of the furnace proper was extended because of the high temperature, and this extension, which consisted of a refractory cover made of lumnite cement and asbestos reinforced with steel, was finally sealed off by a tube made of pipe fittings.

During the experiments the melt was observed through a glass window in the top tube, and conditions were favourable for temperature measurements with an optical pyrometer. A plug on the side of the tube was removed when the slag materials were introduced, and a small section of tube located just below this plug served as a centring device for charging, being exactly over the centre of the rotating liquid crucible, and accordingly directed the fall of the charge to the centre of the cup of metal. Nitrogen gas, containing approximately 0·5% of oxygen, was admitted through a tube opposite this plug, and was allowed to escape through a pipe in the top of the table. The rate of gas passage was controlled and a positive pressure maintained at all times to prevent an infiltration of air.

In carrying out the metal-slag reactions, the preparation of slag constituents in a form suitable for charging into the rotating metal proved to be a critical phase of experimental procedure. It was found, after experiencing some difficulty, that pure powdered silica and pure calcined calcium carbonate could be briquetted after moistening by compressing in a steel cylinder. Cylindrical briquettes were dried and kept at a temperature of 600° C. until ready for use. Although the powdered components were intimately mixed the briquettes melted slowly, and briquettes, especially the most basic ones, floated on the surface of the rotating molten steel before melting, and had a tendency to move up the sides of the molten crucible until they contacted the alundum crucible and were rapidly absorbed. This resulted in fluxing, and the formation of low melting constituents, which soon produced a hole in the side wall of the crucible.

First experiments showed that the melting of a briquette was facilitated by absorption of iron oxide from the metal, and further tests showed that the speed of melting could be increased by dropping small pieces of magnetite on the briquette. In the procedure finally adopted magnetite was added to briquettes of all basicities. As all the tests reached equilibrium with iron oxide in the slag, the amount used to facilitate melting was not critical, and in most cases 5% Fe_3O_4 was added to the lime-silica powders before briquetting.

The synthetic slag mixtures which were added to the molten steel containing appreciable amounts of manganese, contained manganese dioxide in a powdered form. A slag mixture used on a bath containing 0·25 to 0·40% manganese contained 5% manganese dioxide, while the mixture used on a bath containing 0·7 to 1·0% manganese contained 10% manganese dioxide. The slags containing 5% manganese dioxide melted satisfactorily, but those containing 10% manganese were difficult to melt.

It became apparent after a few tests that magnetite was better than manganese dioxide in forming a liquid slag, and that fluid oxidising slags or those containing iron oxide formed more readily and reduced the number of failures due to contact between the slag and refractory crucible.

This was most pronounced in the basic slags to which manganese dioxide had been added, so that most of the tests were made with slags containing iron oxide.

In tests made with metal deoxidised with silicon and carbon, the initial slag mixture contained 5% Fe_3O_4. These slags melted readily and the iron oxide was rapidly reduced from the slag, but not before the lime and silica had reacted to form liquid calcium silicates. With rather

1 E. P. Barret, W. F. Holbrook, and C. E. Woods. Trans. Amer. Inst. Min. and Met. Engs., 1939, vol. 135, pp. 73–84

2 F. W. Scott and T. L. Joseph. *Metals and Alloys*, 1942, vol. 15, No. 3, pp. 414–419.

a thin layer of liquid slag in contact with the metal bath, equilibrium was quickly established.

To obtain data representing equilibrium conditions, with respect to sulphur distribution between slag and metal a technique was developed by which slags could be removed from the surface of the metal at the existing temperature and chilled instantly to prevent any change in composition. The first procedure experimented with for removing the molten slag was to chill it on the pointed end of a heavy copper cylinder, but this method was difficult to operate because the view into the crucible was obstructed by the copper cylinder. With the view partially cut off and the crucible in motion, it was difficult to just touch the slag and not make contact with the steel. Also, if the cylinder was inserted too slowly, the top was heated to such a temperature that the slag would not chill completely and would drop off while being withdrawn from the furnace.

A second and better method of removing the slag was developed by removing it with suction into a small graphite cup, where it was instantly chilled. The graphite cup was tightly fitted into the end of a $\frac{3}{8}$ in. diameter tube connected to a vacuum pump, and when the cup came into contact with or in very close proximity to the molten slag it was instantly drawn into the cup and chilled. The slag remaining on the bath was not disturbed, and the small diameter of the tube and the cup allowed a good view of the slag during the operation, and as a result very few melts were lost because of inability to remove the slag and chill it to prevent any reaction with the metal below the temperature of the test.

Electrode Holders

ARC welding is an important operation in the present production drive. It is therefore important that the equipment employed in performing this operation should permit fast welding with the minimum of fatigue to the operation. To obtain maximum efficiency, care must be exercised in the choice of electrode holders, and special advantages in this direction are claimed for the "T" type holder manufactured by the Lincoln Electric Co.

The special feature of this holder is that the design permits the electrode to be used down to the end of the coating, which reduces the size of steel ends, thus saving metal, electrodes, and time in replacing electrodes. This holder is light and well balanced; the electrode is gripped in the jaws by a powerful spring, enabling the operator to concentrate on welding, since it is not necessary to grip a lever to keep the electrode in place.

A woman welder at work on a light-gauge vertical welding job, using a 14 oz. holder.

Women welders are being increasingly employed on all types of light-gauge work, and the "ST" holder may be used with ease for vertical welding, as in the accompanying illustration. Weighing only 14 oz., this holder considerably reduces operator fatigue. For the use of higher currents and larger electrodes another design of holder, known as the "HT" type, is recommended. The jaws in this holder are faced with special metal, providing a high electrical conductivity and maximum wear resistance under heat. These holders are briefly described in a brochure which can be obtained from the Lincoln Electric Co., Welwyn Garden City, Herts.

A Magnetic Method for Testing the Condition of Hardened and Tempered Finished Steel Components

THE magnetic testing of finished components is used to separate defective work from useful products with damage to the products in the testing procedure. Two methods may be adopted, the first may be used to find out whether the component is free from small irregularities such as scratches, slag inclusions, segregations, etc.; the second, to test the composition of the material and its properties such as tensile strength, hardness, etc.

While the first group has been developed in two directions, viz., making visible the course of the magnetic power lines by magnetic powder, or by measuring the magnetic field, and is now used in practice; the second group is rarely considered and results serviceable in practice have scarcely been reported. In an effort to fill the gap in the latter group an investigation* has been carried out by H. Lange.

Methods employed in this second group measure the magnetic properties of the components in order to draw conclusions regarding other, mostly mechanical, properties. Since the mutual relations between the two kinds of properties are not yet completely known, it is commendable to carry out the magnetic testing as comprehensively as possible. That is best done by recording the hysteresis curve, from which individual figures of coercive power, remanence, etc., can be taken direct.

If it is intended only to separate defective from good components, it is sufficient to determine differences against a standard component of known good quality. Thus, the problem is concerned with recording the difference in inductive capacity between two specimens, one the standard component, and the other with which comparison is sought, by plotting against the strength of the magnetic field by which both have been influenced simultaneously.

In principle this problem is solved by the Siemen's ferro-meter in conjunction with a co-ordinate recorder—i.e., two highly sensitive moving-coil mirror galvanometers recording to ordinate at 90°. Experiments with this apparatus have shown that the hardness properties of heat-treated components can be determined by very weak magnetic fields.

Relying on the experiences arising from these experiments, a simple apparatus can be developed for checking the hardness of quenched and tempered components by a magnetic method, which could be considered suitable in practice, and Lange gives a detailed report of tests carried out by means of such an instrument.

* Mitteilungen aus dem Kaiser-Wilhelm Institut für Eisenforschung zu Düsseldorf, vol. xxi, No. 6.